

# TOPICS OF THE MONTH

## Fuels of the future

CHEMISTS and chemical engineers interested in the ocean as a source of chemicals have probably never considered sea water as a serious competitor to petroleum in supplying the energy needs of the future. Yet, as an American nuclear scientist points out, sea water could be a better fuel than petrol as science learns eventually to harness thermo-nuclear reaction. To back up this statement, he points out that deuterium or "heavy hydrogen" in ordinary water packs more energy "than you could obtain in the equivalent volume of gasoline". If you can visualise all the oceans of the world turned into gasoline, he says, you have a rough idea of the potential energy reserve.

This aspect was discussed by Dr. C. A. Larsen—until recently director of the Atomic Energy Commission's Oak Ridge Laboratory—at the Nuclear Engineering and Science Congress which was held in Cleveland, Ohio. Looking at the energy situation from another angle, H. R. Batchelder and H. W. Nelson, technologists at Battelle Institute in the U.S. foresee the production of synthetic fuels in special areas in the near future, and believe that synthetics will begin to come into general use within the next decade. In a paper presented before the American Institute of Mining and Metallurgical Engineers and the American Society of Mechanical Engineers, they point out that atomic power production will not necessarily alter the picture. According to Atomic Energy Commission estimates, atomic power may reach the equivalent of 2,500 trillion B.Th.U. per year in 1975, but this would amount to less than 5% of America's predicted total energy needs. Also, even if atomic power should fulfil the most optimistic expectations, there would still be greatly increased need for liquid fuels for special applications, such as for automotive power.

On the basis of known U.S. oil reserves and estimates of imports, it is calculated that the peak use of petroleum as an energy source will have been passed before 1975. Coal will then be the most likely source for the synthesis of liquid fuels. As the production of petroleum and natural gas declines, the Battelle technologists expect the production of coal to rise proportionately.

In the U.K., where coal and not petroleum fills the picture at the moment, there is some latent anxiety about the future of coal mining in view of atomic energy plans. To put the matter in perspective Dr. J. Bronowski, director of the N.C.B. Central Research Establishment, has recently summed up the prospects for coal and shows the trend which he thinks will be of major importance to the mining industry for the next 30 years. In a lecture delivered before the Association of Mining Electrical and Mechanical Engineers he points out that atomic energy does not challenge the use of coal, but challenges the use

of coal in making electricity. There is an intelligent future for coal which depends on rejecting the historic role of coal as a poor relation, tied mainly to the making of cheap power, and instead takes advantage of the deeper nature of coal as a chemical raw material.

It was in this way that the oil industry raised itself from providing kerosene for lamps to the major chemical industry it is to-day. Coal can follow the same progress, step by step, as it is freed from the clutch of the famine for power which holds it now. One implication, Dr. Bronowski points out, dominates all others: that the industry, from being mainly an extractive industry, should become in an important sense a processing industry. The changes he foresees will make two demands from engineers: a new conception of rapid and low cost mining of small coal, and a new interest in cleaning, processing, and other chemical engineering plant at the pithead.

## Britain's chemical industry expands

STILL increasing production, increasing exports and increasing manufacturing facilities are indicated by the latest figures available for Britain's chemical industry. The official index of production shows that whereas the average level of production within the chemical and allied trades for the first six months of 1954 was 66% higher than that for the whole of 1948, the average for the first six months of 1955 showed an increase over 1948 of 79%. The comparable increase for the whole of the manufacturing industry was 43%.

Commenting on these figures in its annual report, the Chemical and Allied Trades Section of the Manchester Chamber of Commerce points to the interesting fact that this production increase within the chemical and allied trades since 1948 has been accompanied by a rise in employment figures of only 17%. It also points out, that, in common with other industries, chemical manufacturers have experienced increased cost of labour, fuel, raw materials and transport, while the dock strike of last year and, to a lesser extent, the rail strike, created problems.

The chemical industry has again improved upon its export performance of previous years. Exports of chemicals as listed in the United Kingdom Trade and Navigation Accounts during the 10 months January/October 1955 stood at £193 million, as against the £168 million for the same period of 1954—an increase of 15%. Re-exports of imported chemicals also increased from £2.7 million to £3.9 million. While these results are remarkable in view of the dock strike, it should not be thought that trade is for the asking in easy conditions of a seller's market. They have been achieved in face of mounting competition from other producing countries and with a continuance of tariff barriers and import restrictions in many important markets.

Imports of chemicals have also increased.

### Big new projects near completion

FURTHER substantial additions to manufacturing capacity will come into operation this year, ensuring that the steady rise in the output of the chemical and allied industries will continue. Of the new projects surveyed in a recent *Financial Times* article, it is noticeable that a large proportion are for the production of plastics, synthetic rubber, and the basic materials that go to the making of them. Among the exceptions are the second unit of I.C.I.'s Terylene synthetic fibre plant and the same company's new chlorine plants. Big increases in chlorine and caustic soda capacity will also be completed by the Murgatroyd Salt & Chemical Co. this year. Other new facilities include extensive additions to Laporte Industries' hydrogen peroxide plant, and the new plant at Stallingborough (doubling the group's output of titanium oxide) which started production recently. Shell Chemicals' £1½-million, 20,000-ton-capacity alkyl-benzene plant at Shellhaven—the first major chemical unit to come into production there—is on the verge of completion as we go to press. Fisons plan to complete, during 1956, the first ethylene diamine plant in the U.K., and will also bring greatly increased capacity of hydrazine into operation.

I.C.I.'s 10,000-ton butadiene copolymer plant will come into production this year, drawing its butadiene from the first butadiene extraction plant at Wilton which is due to start up in the spring. The Plastics Division will complete a new PVC plant which will raise total capacity to about 40,000 tons compared with 25,000 to 30,000 tons in 1955. The 200-ton PTFE plant will also be completed this year. In the middle of the year Billingham Division hopes to bring into operation a second olefine plant and an extension of its glycol plant. The General Chemicals Division expects to bring substantial additions to its chlorine producing plants into operation which will make material available for extensions to the monochlorobenzene and sodium cyanide plants at Billingham and for other purposes. When the scheme for expanding these plants was first launched total expenditure was estimated at £22m.

Monsanto Chemicals will complete a major extension of polystyrene capacity at its Newport works this year, mainly for the production of high-impact-grade polystyrene. The 4,000 tons synthetic rubber plant which has been built at Newport at a cost of about £500,000 will shortly come into operation. It will produce a wide range of styrene/butadiene copolymers. Another large Monsanto plant which is due to start up this year is the maleic anhydride plant at Newport, while at the Ruabon works, a new plant will come into production towards the end of the year which will give expanded facilities for the large range of rubber chemicals manufactured there. The largest project at Ruabon is the long-term programme for the expansion of phenol production, but only part of this is expected to come into production during 1956.

The big new styrene monomer plant of Forth Chemicals Ltd., at Grangemouth, is due to come into operation shortly, while, nearby, British Petroleum

Chemicals is engaged in a series of major extensions. At Fort Dunlop the Dunlop Rubber Co. hopes to complete its £½-million, 2,000-ton experimental synthetic rubber plant during the year.

Albright and Wilson plans to spend about £1 million this year on a number of projects including extensions to the oil additives plant and new plants for making organic phosphorous compounds used in the plastics industry and in the new *Proban* anti-flame finish for textiles and sodium hypophosphite used in the new *Kanigen* chemical nickel plating process. This sum does not include expenditure by Marchon Products and Solway Chemicals at Whitehaven which will amount to about £½m. and will be applied mainly to extensions to detergent materials.

### Russia's new industrial drive

RUSSIA has announced an all-out drive to "overtake and surpass" the Capitalist West in industrial, atomic and agricultural development. The challenge came in a new five-year plan issued by the Soviet Communist Party covering the years 1956-1960 inclusive.

Targets for the five years from 1956 until 1960 (inclusively) call for a steady expansion of heavy industrial output, until by the end of 1960 a "norm" is reached of 53 million tons of pig iron, 68.3 million tons of steel, 593 million tons of coal and 320,000 million kilowatt hours of electrical energy. The target for electricity represents almost a 100% increase over 1955.

Western industries can be sure that this will be accompanied by increased production of heavy and other chemicals, as well as greater activity in chemical engineering and related fields.

### Isotopes in new pipeline application

AN interesting application of tracer technique proved to be the answer to a monitoring problem posed by an oil company operating in the Middle East. The company's engineers wanted a means whereby a change in the batch of oil passing through the pipeline between the oil field and the tank farm a hundred miles away could be signalled at just the right moment to allow the two batches to be piped into separate tanks.

A solution was found as follows. On switching oils at the well end of the pipeline, the operative inserts a plug containing a small quantity of radioactive materials. This is then carried down by the oil flow, and on approaching the tank farm, is 'detected' by a monitor sited some distance out in the desert. A warning is then automatically given to the valve operator and at the correct moment he is able to redirect the flow to the appropriate tank.

This new application of isotopes was described recently in the magazine of the 600 Group, which adds that the monitor, and the alarm system to which it is connected had, incidentally, to be designed to operate in intense heat and at the same time be proof against the inquisitive probing of passing Arabs.

## World titanium production increases

A SHARP rise in the production of titanium in 1956, to meet a quickly rising demand, has been forecast in America. Current U.S. Government estimates show probable 1956 requirements for mill products ranging from 4,000 to 5,000 tons, thereby indicating that fabricators will be under extreme pressure to more than double their record 1955 activity.

In a year-end review of the titanium industry, an official of the Titanium Metals Corporation of America comments that fabricators had been expanding mill facilities in preparation for a rush of high-priority demands. No significant delivery lags were anticipated. The value of titanium mill products sold in 1955 was estimated at \$6 million, with the sales of sponge—the pure intermediate metal from which mill products are made—accounting for an additional \$30 million.

The Japanese also expanded their output in 1955, exporting most of their production to the U.S. These sponge imports were estimated at 500 tons, compared with 255 tons during the previous year.

Britain's sole producer, Imperial Chemical Industries, is stated to have achieved virtual capacity operation of 1,500 tons p.a. late in 1955, and this output was absorbed by rapidly expanding aircraft requirements in the U.K. I.C.I.'s big new project for the fabrication of titanium was referred to in our December 1955 issue, and now Laporte Titanium Ltd., who, as mentioned in a preceding note, have recently brought a new titanium oxide plant into production, are planning to increase the capacity of their Stallingborough plant by a further 12,000 tons a year, to give a total annual output of 30,000 tons p.a. of titanium oxide. This marks the beginning of phase No. 3 for their plant, which was built in 1953 and has already been considerably enlarged.

## Comical Engineering Situations



"LUMME — MOTHS!"

## Air pollution in the U.S. chemical industry

ABATEMENT of air pollution is a subject which is getting more and more attention in the United States, as is evident from the fact that the chemical industry there is now spending at least \$40 million yearly on air pollution efforts. To many observers, the fast growth of the American chemical industry has hidden the real advance made in individual cases of pollution abatement. Los Angeles presents an excellent example of how difficult it is for improvement in individual cases to compensate for the rapid increase in the number of potential pollution sources.

This problem was pointed out at a meeting of the U.S. Industrial Hygiene Foundation, in Pittsburgh, by Mr. M. F. Crass, Jun., secretary of the Manufacturing Chemists' Association, who gave some examples of just how expensive air-pollution abatement measures can be in some cases. In one instance, an expenditure of \$340,000 was required to install a scrubbing and stripping unit in an existing Texas sulphuric acid plant to reduce waste gases going to atmosphere from .4% SO<sub>2</sub> to .03% or less and at the same time neutralising scrubber liquor to avoid stream pollution. But, in this particular case, it seems, the recovery plant provided a 20% increase in acid production. Such economic return is the exception rather than the rule.

Another case, involving the disposal of antibiotic waste, is something of a classic, since the factors involved, while typical in many respects, represent the lengths to which management will sometimes go to in its efforts to be a good neighbour. As well as the antibiotic, waste products were produced which involved a solid residue and a liquid effluent, both of which gave forth a brewery-like aroma which, while not unduly obnoxious, nevertheless created complaints. Efforts to achieve abatement included underground burial and even the construction of a special multi-mile sewer, but the odour persisted. It was only after three years of trial and error that a solution was found. Total cost of eliminating the odour and disposing of the liquor effluent—\$1 million.

The wisest pollution abatement policy, of course, is for firms to consider the disposal of all wastes as an integral part of any new plant or process. As Mr. Crass points out, it is just as much a job of research to develop good methods of controlling air and water contamination as it is to develop new products and processes.

## Corrosion of condenser water boxes

THE corrosion of water boxes of large boiler condenser installations is a major problem. These water boxes are usually constructed of cast iron, and the condenser tubes connected to the water box are usually of Monel, stainless steel or some corrosion-resistant alloy such as cupro-nickel. The galvanic effects set up in such a system, where the cast iron serves as an anode to protect the tubes, are enough to cause serious corrosion of the cast-iron housing to the point where it must be replaced periodically.



This is especially true in installations that employ sea water as the coolant in the condenser. Application of a coating to the water box, to reduce the protective current, retards its corrosion.

Several industrial concerns recommend coatings for painting the interior of the water boxes. One supplier recommends a Thiokol-type synthetic rubber coating, to be applied  $\frac{1}{8}$  in. thick.

Discussing this subject in the January issue of *Corrosion Technology*, B. G. Brand pointed out that the Dow Chemical Co. has made a study of this problem at its Freeport, Texas, plant, where sea water is used to cool ethylene oxide. Dow found very few coatings that have an appreciable life under these conditions, and those that have a significant life usually develop pinholes, which are sites for corrosion accentuated over that found in bare metal. Moreover, use of a coating of the rubber type removed the cathodic protection usually relied on to protect the tubes by sacrifice of the cast-iron water box. This resulted in undue corrosion of the condenser tubes—much more costly than water-box corrosion.

Use of cylindrical magnesium anodes in the coated water box effectively stopped corrosion of both tubes and water box. The tests have been under way at Freeport, Texas, for about five years, with no visible signs of corrosion. This installation has a carbon steel water box, Monel tube sheets and cupro-nickel tubes.

#### From surgeon to soda-maker

ALTHOUGH the Leblanc process for soda production proved one of the very foundations of the British chemical industry, how little tribute has been paid to its originator, who died by his own hand just 150 years ago. In our chemical history texts we may read much of Gamble, one-time minister of the kirk of Enniskillen, who gambled his capital as alkali-maker; the Gamble who rode to his St. Helens works in his broad-brimmed hat and broadcloth spotted with acid. The works were in such rural surroundings that pheasants might roost in the warm sheds while a stag escaping from the St. Helens hunt was chased around the carboys. We can learn, also, of Muspratt, that military man of the Peninsular War, who went to Liverpool and then to Widnes to work the Leblanc process, though HCl fumes blasted the countryside and turned Widnes into a waste. Yet of the actual creator of the process, how few facts are given. The anniversary of his death provides a convenient point from which to look back on his struggles.

Nicolas Leblanc, born south of Orleans, began as a pharmacist's apprentice but on turning to surgery he won such renown that he became surgeon to the Duke of Orleans. It was this same duke who found the capital to finance Leblanc's industrial plant at St. Denis, and who was executed in that merciless Revolution which brought confiscation of the factory and the end of Leblanc.

To appreciate the value of the process or technique of this pioneer, it is necessary to look back at previous soda-making efforts, such as that of Malherbe at the

#### A SMALLER 'C.P.E.' THIS MONTH

OWING to the dispute between the British Federation of Master Printers and the Typographical Association (one of the printing trade unions) we have been reluctantly compelled to reduce the number of pages in this issue of *CHEMICAL & PROCESS ENGINEERING*. This reduction makes itself most evident in our regular monthly feature, 'What's News about Plant, Equipment, Materials and Processes' (pages 75 to 78) which is half its usual size.

The dispute is completely outside our control. As soon as a settlement has been reached we shall be glad to resume normal publication.

St. Javel works. In this, a mixture of sodium sulphate with carbon also included metallic iron, the latter yielding iron sulphide while the sodium in contact with air, moisture and carbon dioxide from furnace gases formed a crude product of sodium carbonate plus hydroxide. It was a curious method in that the resulting blocks of fused product were allowed to stand in moist air to fall to a crumbly mass of soda as final product. There were other experimental efforts with sodium sulphate plus coal as reaction mixture, with salt plus copperas, or salt plus lime heated together.

All these bore no comparison in efficiency with Leblanc's successful process, described in his patent of 1791 and in a memoir published in the *Annales de Chimie* in 1797. The conversion of surgeon Leblanc to soda-manufacturer had come when the Académie des Sciences offered a prize for some method of producing alkali other than from natural sources such as barilla. He prepared sodium sulphate by Glauber's process, the HCl evolved being absorbed in ammonia liquor—a procedure not only dealing with the nuisance of acid fumes but yielding ammonium chloride as by-product. Whether Leblanc used calcium carbonate after heating of Margraf's preparation of calcium sulphide from gypsum and powdered coal can only be guessed. Certain it is that his grinding together sodium sulphate, chalk and charcoal, his heating this mixture at first in crucibles and later by raking over the bed of a reverberatory furnace all proved the first successful soda process in history. Yet in 1794 the patent was cancelled by a special commission soon after the Revolution had taken its toll of progress, the commission betraying utter ignorance of chemical matters in suggesting that such processes as that of Malherbe were more likely to succeed. Although Leblanc claimed damages when his factory was confiscated, although he continued to work unpaid at other factories, the end came in 1806. His extreme poverty and the irony of a ruined factory returned to him when no capital was available to set it going, all proved too much. This pioneer in soda-making, who takes his place along with Solvay in industrial chemical history, shot himself in a pauper institution. Only his name lived on in the Leblanc process which just a century ago, in Lancashire and Worcestershire, was in its heyday.



# Hydrazine as an Oxygen Scavenger

By S. R. M. Ellis, PH.D., F.R.I.C., A.M.I.CHEM.E., and C. Moreland, B.Sc.

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In March 1955 we published an article by Dr. Ellis in which he discussed some aspects of boiler feed water treatment. The latter half of this article was concerned with the use of hydrazine as an oxygen scavenger in place of sodium sulphite. In the present article, some recent experiments on the use of hydrazine as an oxygen scavenger are described and the results are presented and discussed.

IN recent years there has been a steady increase in working temperatures and pressures of steam boilers and this has resulted in a need for high-purity boiler feed water, for a low solids content and a low oxygen concentration in the feed water decrease the amount of blow-down, eliminate scale formation and reduce corrosion.

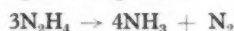
For the removal of oxygen from boiler feed water the most common practice has been to use a mechanical deaerator, followed by a chemical deoxygenator such as sodium sulphite. Although sodium sulphite effectively reacts with oxygen, recent developments show that at high temperatures and pressures the sulphite breaks down in the boiler to sulphur dioxide,<sup>1</sup> thus giving rise to fresh corrosion problems. Much attention has therefore been directed towards the use of hydrazine<sup>2-10</sup> as an alternative chemical deoxygenator.

Hydrazine does not increase the total dissolved solids in the boiler and is a strong reducing agent capable of reacting with oxygen to give nitrogen and water as reaction products. The general reaction of hydrazine with dissolved oxygen is shown by the following equation:



In practice the reaction is not so simple, but this equation nevertheless describes the net result of the removal of oxygen.

At temperatures in the boiler above 350°F., excess hydrazine is partly vaporised and partly decomposed according to the equation:



Because of this loss of hydrazine it is desirable that the reaction between hydrazine and dissolved oxygen should be largely completed before the feed water enters the boiler.

The experimental work of Gilbert,<sup>11</sup> Wickert<sup>12</sup> and Audrieth<sup>13</sup> shows that at and near the saturation concen-

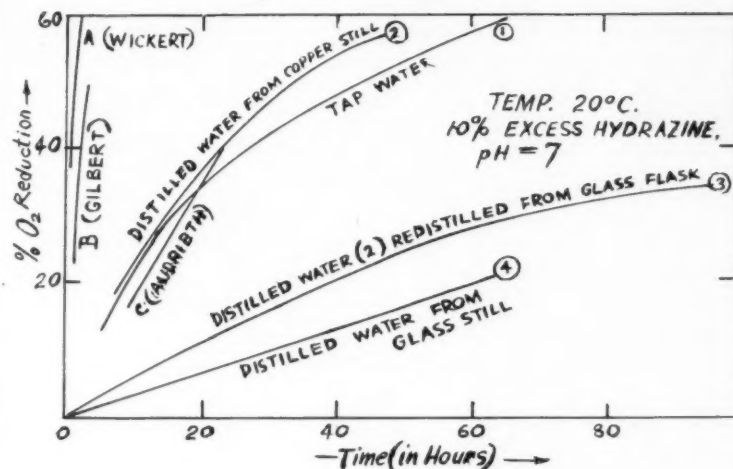


Fig. 1. Results for hydrazine oxygen reaction using water from different sources.

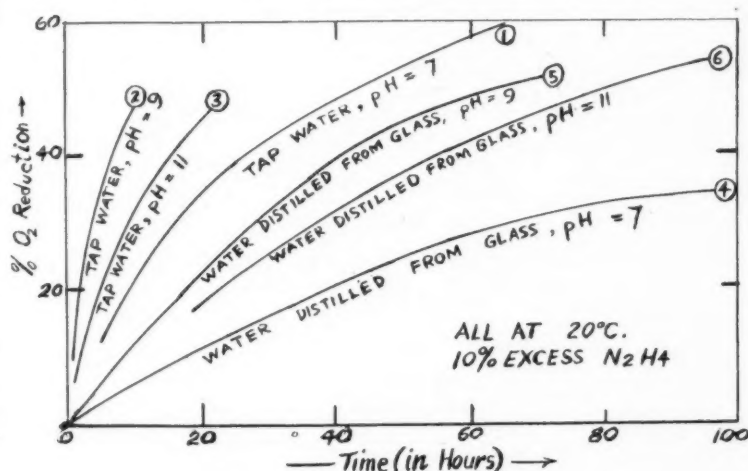


Fig. 2. Effect of varying pH value of water.

trations of oxygen the reaction between hydrazine and oxygen is not instantaneous. It is important, therefore, that rate of reaction data should be available for different feed conditions and varying temperatures. As a first step, reaction rates have been deter-

mined at a high concentration of oxygen in the feed water, because of the greater convenience and rapidity with which it is possible to investigate some of the numerous factors influencing the rate of reaction between hydrazine and oxygen.

## Experimental procedure

All experimental work described in this paper has been carried out at oxygen concentrations of between 0.1 and 8 mg./l. The course of the reaction was followed by analysis for oxygen content rather than for hydrazine. The direct estimation of oxygen was considered preferable to the determination of the hydrazine concentration, which may vary by other reactions than that with oxygen.

The analytical method adopted was basically that of Rideal and Stewart,<sup>14</sup> where the hydrazine is first removed by potassium permanganate and the oxygen estimated by the Winkler method. In later experiments, bromine was used to destroy the hydrazine, as suggested by Wickert and Ipach.<sup>15</sup>

Initially, experiments were done under static conditions, the aerated water and hydrazine being allowed to stand in glass flasks completely filled with water and periodically analysed for oxygen. Except where otherwise stated, all experiments were done at 20°C. The water used was distilled in glass equipment and to this distilled water was added tri-sodium phosphate to bring the ( $\text{PO}_4$ ) concentration to about 15 p.p.m., and the pH to 9 or 10. A small excess of hydrazine (about 10%) over the theoretical amount was used. All analyses were carried out in duplicate.

In later experiments, water containing hydrazine was allowed to flow through a column packed with a

catalyst and analysed for oxygen as it emerged from the bottom.

## Presentation of results

The results are presented graphically as plots of percentage of dissolved oxygen removed against reaction time. The resulting curves have been smoothed and experimental points omitted for the sake of clarity. In practice, a scatter of points was obtained as shown in Fig. 4.

The initial oxygen concentration was just below the air saturation value, i.e. between 6½ and 8 mg./l. at 20°C., and somewhat lower where higher temperatures were used.

In the work that follows, the effect of varying the following factors on the course of reaction was investigated:

- (a) purity of the water;
- (b) pH of the water;
- (c) excess of hydrazine;
- (d) temperature of the water;
- (e) the presence of metallic ions; and
- (f) the presence of heterogeneous catalysts.

(a) **Purity of water.** Fig. 1 shows the results for the reaction using water from different sources. The reaction is very slow, much slower than found by Wickert<sup>12</sup> or Gilbert<sup>11</sup> and a little slower than found by Audrieth.<sup>13</sup> The results of these workers are plotted in Fig. 1 for comparison.

The rate of reaction can be seen to be very sensitive to the type of water

used and becomes slower as the purity of the water increases. The rate is seen to be extremely slow in the case of water distilled from glass vessels.

Subsequent work was directed towards increasing the rate of reaction by various means.

(b) **pH value of the water.** The results with different pH values (both tap water and water distilled from glass vessels) are shown in Fig. 2. For both, a marked increase in rate is seen when the pH is increased from 7 to 9. With further increase in the pH to 11, the rate falls off to an intermediary value. This is in agreement with the findings of Gordon,<sup>16</sup> who found hydrazine to be most effective as a reducing agent in the pH range 9 to 10½. This fact is important, since most boiler feed waters are controlled to this pH range.

However, the rates are still very slow, and further modifications are necessary to increase them.

(c) **Excess of hydrazine.** The method of analysis is applicable only to a maximum of 300% excess hydrazine over the theoretical amount required for complete deoxygenation. The effect of excess hydrazine on water of pH = 7 and pH = 9 is shown in Fig. 3. At a pH of 7, even with 300% excess hydrazine, the reaction is still very slow. At pH = 9 the increase in reaction rate observed on adding 200% excess of hydrazine is in reasonable agreement with that expected by theoretical considerations

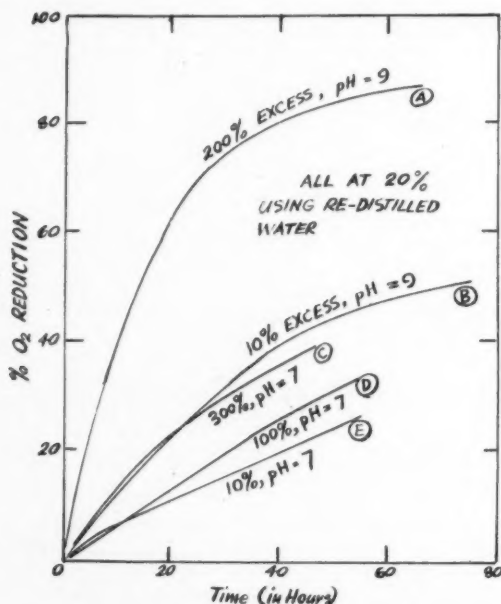
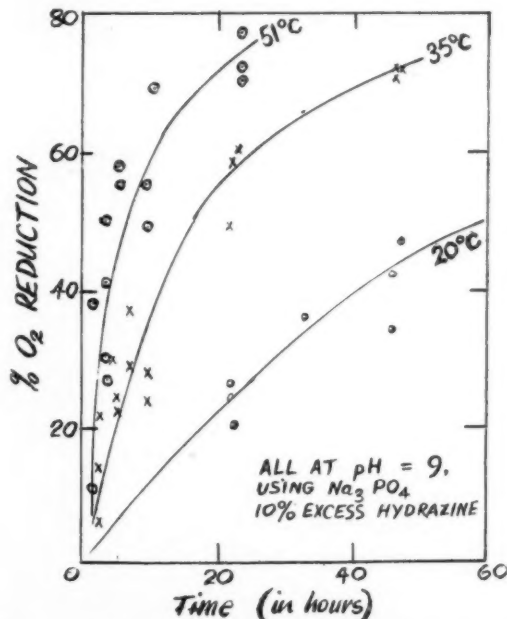


Fig. 3 (Left) Effect of varying excess hydrazine. Fig. 4. (Right) Results obtained at different temperatures.



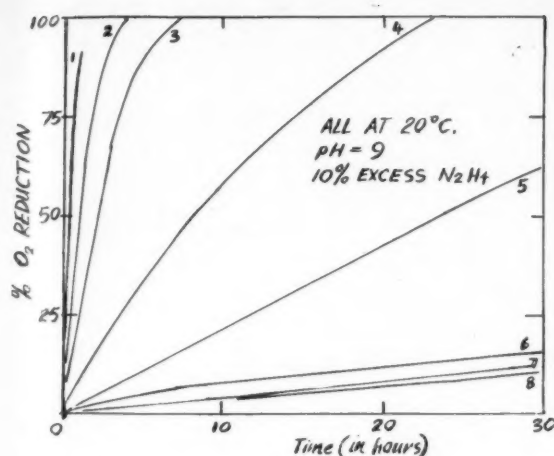
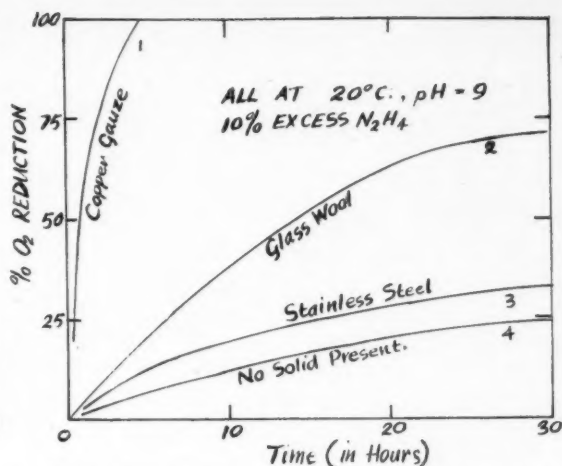


Fig. 5 (Left) Effect of metallic ions on hydrazine oxygen reaction. Curves are for (1) 5 p.p.m.  $\text{Cu}^{++}$ , (2) 1 p.p.m.  $\text{Cu}^{++}$ , (3) 1 p.p.m.  $\text{Co}^{++}$ , (4) 5 p.p.m.  $\text{Mn}^{++}$ , (5) 1 p.p.m.  $\text{Mn}^{++}$ , (6) 1 p.p.m.  $\text{Fe}^{++}$ , (7) 1 p.p.m.  $\text{Cr}^{+++}$ , (8) 1 p.p.m.  $\text{Ni}^{++}$ . Fig. 6 (Right) Curves showing the effect of various solids when serving as catalysts.



assuming the reaction to be of the second order.

(d) **Temperature of the water.** The reaction was followed at 20°C., 35°C. and 51°C., and the results obtained are presented in Fig. 4. Over this small temperature range there is a steady increase in rate with temperature. The rate is approximately doubled for a 10°C. rise in temperature, as for many chemical reactions.

The rate is still slow, as even at 51°C. only 60% of the oxygen has reacted after 10 hr. Assuming the reaction rate to be doubled for a 10°C. rise in temperature, it is calculated that at higher temperatures of the order of 170°C. the time of reaction would be only a few seconds.

(e) **Presence of metallic ions.** A number of experiments were conducted with traces of soluble metal salts added to the reaction flasks to see if these would catalyze the reaction. The metals used were  $\text{Cu}^{++}$ ,  $\text{Fe}^{++}$ ,  $\text{Co}^{++}$ ,  $\text{Ni}^{++}$  and  $\text{Cr}^{+++}$ , and  $\text{Mn}^{++}$  added as the sulphate in concentrations giving 1 p.p.m. of the metallic ion. Usually a haziness developed in the reaction flasks after the addition of hydrazine, which was presumably due to the formation of an insoluble reduced complex.

The results are shown in Fig. 5. The presence of 1 p.p.m. of either copper or cobalt ions gives a great increase in the rate of reaction; the reaction is completed in 4 hr. at 20°C. A concentration of 0.5 p.p.m. of Cu gives a rate of reaction similar to that of Curve 3, Fig. 5, for 1 p.p.m. of Co.

Curves 1 and 4 show that increasing the copper and manganese content to 5 p.p.m. further increases the rate of

reaction, compared with Curves 2 and 5 at 1 p.p.m. Although not shown on Fig. 5, increasing the Fe content to 10 p.p.m. also increases the rate of reaction.

(f) **Heterogeneous catalysts.** In this series of experiments, solid insoluble material was immersed in the reaction solutions so as to present a relatively large area to the reactants. Some of the results are shown in Fig. 6.

Copper gauze was seen to increase the rate of reaction to a very large extent, the reaction being complete in about 5 hr. at 20°C. On the other hand, stainless-steel gauze had practically no effect on the rate of reaction.

The effect of glass wool was most interesting, in that it gave a considerable increase in the rate of reaction. It suggests that the reaction is catalysed at a surface and that the increased rate was due to the large increase in area available. The results using copper and stainless-steel gauzes show the reaction to be sensitive to the nature of the surface. This conclusion was also confirmed by experiments which showed that active carbon and ion-exchange resins could also act as catalysts.

Gilbert<sup>11</sup> postulates a surface reaction and Zimmermann<sup>3</sup> also believes the reaction to be a surface one. Audrieth<sup>13</sup> has pointed out the sensitivity of the reaction to the nature of the surface of the reaction vessel.

#### Carbon column

In this series of experiments, water with hydrazine was allowed to flow down a column packed with active carbon, and the oxygen content of the

treated water was determined. The column used was a 1-in.-diam. glass tube, packed with carbon of 12 to 25 mesh. Preliminary experiments showed that different grades of active carbon gave similar results.

The time of contact of the water with the carbon was varied by changing the flow rate and by changing the length of the column. The solution was fed to the column at temperatures in the region of 20 and 40°C., the initial oxygen concentration at the former temperature being about 8 mg./l. and at the latter about 6 mg./l.

Approximately 40% excess of hydrazine was used in all but one experiment. Tri-sodium phosphate was added to all solutions before starting, to raise the pH value to 9 to 10.

The results obtained are presented in Table I.

The carbon has a very great effect on the rate of reaction. These preliminary experiments indicate that the reaction is completed in under 90 sec. at 20°C. and in 9 sec. at 42°C.

The addition of a trace of copper sulphate to the water did not increase the activity of the carbon. Recent information from Germany<sup>3</sup> and from Italy<sup>5</sup> states that carbon is used as a catalyst for the oxygen/hydrazine reaction in the deoxygenation of boiler feed waters.

#### Copper column

A similar series of experiments were carried out using a glass column of 1 in. internal diameter packed with bright copper turnings. In each case 55 cm. of packing were used and the time of contact was varied by altering the flow rate down the column.



As before, a 40% excess of hydrazine was used, and tri-sodium phosphate was added to adjust the pH to 9 to 10.

The results appear in Table 2.

The copper turnings are seen to catalyse the reaction, but their effect is less than that of active carbon.

### Discussion and conclusions

The reaction between hydrazine and water-dissolved oxygen is not a simple one, and is very sensitive to traces of impurities and to the nature of any surface present.

The rate of reaction is increased on increasing the pH value to 9 to 10, but with a further increase in the pH value the rate declines.

Increases in the amount of excess hydrazine, or in the temperature, give an increased rate of reaction.

The presence of 1 p.p.m. of copper or cobalt ions gives a very large increase in the rate of the oxygen/hydrazine reaction; a solid metallic copper surface will also catalyse the reaction. This fact, together with the effect of glass wool and the very great effect of carbon, leaves no doubt that the reaction can be catalysed by a solid surface and, in fact, may be largely a surface reaction.

The use of active carbon in a packed column gives a very large increase in the rate of reaction, and this has been the most promising catalyst so far found for the reaction.

It would seem, therefore, that in some cases where hydrazine is employed in the chemical deoxygenation of boiler feed waters a catalyst might effectively be used to speed up the reaction. This is particularly so where the temperature in the feed line is

low and where a minimum excess of hydrazine is required.

Passing the feed water with hydrazine over a carbon bed would greatly accelerate the reaction. It has been recently reported in the literature that carbon beds are being used successfully at one Italian power station.<sup>5</sup>

From these preliminary results it is obvious that the reaction between hydrazine and oxygen in water solution is not a simple homogeneous bimolecular reaction. Further work is proceeding on the use of hydrazine in the treatment of boiler water.

The authors acknowledge the help received from Messrs. Whiffen & Sons Ltd., who sponsored the work.

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Table 1. Results of Experiments Using Carbon Column

Length of column, cm.	Temperature, °C.	Rate of flow, cm./sec.	Estimated contact time, sec.	O <sub>2</sub> content of water emerging, mg./l.	% O <sub>2</sub> removed
100	20	0.09	600	0.1	>98
100	20	0.55	90	0.1	>98
29	16	1.67	9	4.8	40
29	20	1.48	10	2.4	70
29	20	0.48	32	0.9	90
29	42	1.67	9	0.15	>97
*29	20	1.38	11	1.0	87
10	42	1.51	3.3	2.0	65
†29	18	1.67	9	3.0	60

\* Using 200% excess hydrazine.

† Water contained 2 p.p.m.  $Cu^{++}$ .

Note: 0.1 mg. O<sub>2</sub>/l. is the lower limit of the method of analysis used.

Table 2. Results of Experiments Using Copper Column

Temperature, °C.	Rate of flow, cm./sec.	Estimated time of contact, sec.	O <sub>2</sub> content of water emerging, mg./l.	% O <sub>2</sub> removed
20	0.12	324	0.4	95
23	0.25	156	4.6	40
38	0.33	120	1.5	75

## British Standards

**Flow cups and methods of use** (B.S. 1733:1955, 3s.). This revision of British Standard 1733 recognises the demand for a wider range of orifice sizes for flow cups, and six sizes of Type A, corresponding to the A.S.T.M. cup, are now included. Further, the number of sizes based on the Ford 'cup viscometer' (Type B) has been increased to four. The tolerance of  $\pm 0.0001$  in. on the orifice diameter of Type A cups has been relaxed to  $\pm 0.0005$  in., the same as for Type B cups. The revision specifies material, dimensions for the cups and their methods of use. Recommendations for the care of the cup, including periodical checking for wear and possible damage, are given. Average flow times of selected mineral oils and instances of the variation of flow times with ambient temperature are also included for guidance.

**Refrigerator oils** (B.S. 2626: 1955, 10s.). For an oil to act as a satisfactory lubricant and/or sealing agent in any refrigerating plant it is essential that it has properties additional to those normally demanded of mineral oils for general lubricating purposes. This British Standard has been drawn up to provide for oils suitable for use in refrigerating systems containing various refrigerants. The range of viscosities of the oils necessary to cover all requirements is rather wide and for convenience it has been arbitrarily divided into four approximately equal parts.

**Cyclohexylamine and ethanadiol** (B.S. 2534: 1955, 'Cyclohexylamine'; B.S. 2537: 1955, 'Ethanadiol' (Ethylene Glycol); 2s. each). Limits are specified for physical properties of the material and for impurities likely to be present, and full details of the methods of analysis are given.

**Rubber joint rings.** B.S. 2494: 1955, 2s. ('Natural rubber joint rings for gas mains, water mains and sewers') replaces B.S. 674 ('Rubber joint rings for water mains and sewers') and B.S. 772 ('Rubber joint rings for gas mains'). The standard specifies six hardnesses of vulcanised natural rubber joint rings for use in mains containing such fluids as town gas, coal gas, water gas, natural gas, water and sewage. Details of the material from which the rings are to be made are laid down, together with requirements for physical properties both before and after ageing. Limits are specified for swelling and water absorption, and sampling and marking requirements are given.

# A Progress Report on EUROPE'S CHEMICAL INDUSTRY

*Chemical production in O.E.E.C. countries increased by 15% in 1954 and a similar rate of expansion is likely throughout 1955. This is the main conclusion reached in a report of the Chemical Products Committee of the O.E.E.C. which has recently been published,\* and which gives a survey of the situation in the chemical industry in 1954 and early in 1955 in Europe, with some reference to the United States. This article summarises some parts of the report and deals mainly with the heavy chemicals and allied sectors.*

THE added value of the O.E.E.C. countries' chemical industry can be estimated at \$5 million to \$6 million in 1954. If this is compared with the gross national product at factor cost for the O.E.E.C. countries combined, which can be estimated at \$174,000 million, it will be seen that the chemical industry represents about 3% of the total.

Whereas the chemical industry's production rose on an average by 15% over 1953, total industrial production increased by 9%. In the United States, chemical production remained stable in 1954, while total industrial production decreased by 8%.

There were some deviations from the general trend in Europe, such as in the soap and tannin industries, which have had their activity reduced owing to competition from new products, while in other branches of the industry, e.g. plastics, the rate of expansion has been far above the average.

The report points out that, in the case of superphosphates, the recent increase in consumption of phosphatic fertilisers, which will no doubt continue even if there are some temporary setbacks, will enable better use to be made of present production capacity, which is still too large. Nevertheless, owing to production costs and the preference of consumers, there is an increasing tendency in some countries to use nitric acid in the manufacture of phosphatic fertilisers, instead of sulphuric acid used in the production of superphosphate. Although the plant required for these 'nitrophosphates' is more expensive, it may be expected that they will absorb at least a part of the increased demand for phosphate fertilisers.

The expected expansion of the world nitrogen industry during the next few years causes apprehension as

to the possibility of disposing of the quantity to be produced. Consumption of nitrogenous fertilisers by O.E.E.C. countries is rising fairly regularly, though at a slower rate than the expansion of production. It would therefore seem that, even taking into account a possible and indeed desirable increase in the use of nitrogen for technical purposes, consumption can hardly be expanded to the extent needed. Thus exports will probably form an even more important outlet than hitherto, particularly exports to underdeveloped countries where requirements can rarely be met by home production. This will, however, only be possible if these countries are in a position to make the necessary purchases.

## Production and trading increases

The greatest increases in chemical industry production compared with 1953 were in Greece (39%), Italy (22%), Norway (21%), the Belgo-Luxembourg Economic Union (19%), Germany (16%) and France (15%).

The labour force employed in the chemical industry in 1954 was about 1.4 million, of which three-quarters were employed in Germany, the United Kingdom, France and Italy. In 1954, investment in the chemical industry in eight countries, i.e. Belgium-Luxembourg, France, Germany, Italy, the Netherlands, Norway, Sweden and the United Kingdom, which account for approximately 80% of total O.E.E.C. chemical production, is estimated at \$775 million. This investment has largely been made in petroleum chemicals, fertilisers, plastics materials and detergents. The report states that a large part of investments has been financed by the industry itself.

The report points out that the level of productivity in this industry is naturally high and that, though exact and comparable figures are not avail-

able, there are indications that much progress has been made in this field during recent years, as is shown by prices for chemicals, which have generally remained stable in spite of the rising costs of raw materials, energy and manpower. Over the last decade and more, price indices for chemicals have generally risen much more slowly than price indices for other industrial products.

Trade in chemicals expanded in 1954 also. Trade between member countries and their overseas territories increased by 19%, or 4% more than production.

Some progress has been made in liberalising trade between O.E.E.C. countries from quantitative restrictions, but it is pointed out that 'despite the fairly high global liberalisation percentage, the expected results will not be achieved so long as a solution has not been found to other problems affecting trade, above all customs tariffs.'

The salient feature of trade with third countries is a rise of 46% in imports of chemicals compared with 1953, while exports only rose by 21%. Even so, O.E.E.C. countries were still net exporters of chemicals in 1954, exports exceeding imports by \$900 million. It is worth noting that the value of O.E.E.C. countries' exports of chemicals to third countries in 1954 was about the same as that of U.S. exports of chemicals (\$1,060 million).

The report pays particular attention to the development of trade with the dollar area, which was in deficit in 1954. This development is due both to a sharp rise in imports and a decline in exports. The Chemical Products Committee considers that, though the development in the case of imports will probably be temporary, no appreciable rise in exports to the dollar area is yet in sight.

The European chemical industry has increasingly to face foreign competition

\* The Chemical Industry in Europe, Organisation for European Economic Co-operation, Paris. Pp. 210, 12s.

—mainly from the U.S.—in view of the fact that in 1954 a larger part of the chemicals produced was sold abroad.

### Basic inorganic chemicals

From the statistics available for soda ash, caustic soda and chlorine, production of chlorine appears to show the largest, and production of soda ash the smallest, increase in 1954, compared with 1953.

**Soda ash.** Output figures provided by seven of the ten producers of soda ash indicate an increase of 10% over 1953. If figures for all O.E.E.C. countries were available, it would be seen that production of soda ash in Western Europe is slightly lower than production in the United States, where output amounted to 4,269,000 metric tons in 1954.

The 1951 level of production has not yet been reached, although statistics available for the first months of 1955 show a steady, if unspectacular, increase, particularly in countries where demand fell off sharply in 1952.

**Caustic soda.** Nine of the 14 producers gave separate production figures for 1954; in all the other five, except the United Kingdom, output is relatively small. In these nine countries, the production of causticised and electrolytic soda reached 1,216,250 metric tons, an increase of 11% over 1953. However, it would appear that O.E.E.C. production is not yet up to the United States' figure of 3,081,600 metric tons in 1954.

Production indices for the nine countries in 1954, 1950 production being represented by 100 as the base, are: B.L.E.U., 144; Denmark, 392; France, 117; Germany, 148; Italy, 160; Norway, 179; Portugal, 237; Sweden, 141; and Turkey, 150.

**Chlorine.** The increased demand for chlorine, mainly for the manufacture of solvents, plastics, pesticides and weedkillers, has stimulated many countries to increase their output, and the total production of nine European countries was 17% over the 1953 figure. United States' output in 1954 (2,628,750 metric tons) was certainly far greater than total production in Western Europe.

Danish production is expected to increase considerably in 1956 to meet demand for the production of hydrogen for oil hardening. Output in this country, and in Portugal and Turkey also, is very small compared with that of the other O.E.E.C. countries.

The general indices for soda ash, caustic soda and chlorine for those O.E.E.C. countries submitting pro-

duction figures, when 1950 = 100, are 117 for 1953 and 131 for 1954. The United Kingdom index for these three chemicals is 101 for 1953 and 118 for 1954.

**Sulphuric acid.** The high level of production of sulphuric acid in 1954 may be taken to reflect the general level of activity in many branches of the chemical industry. Apart from the smaller producers (Austria, Greece and Portugal), whose output has risen considerably over the past five years, the largest increase will be found in Germany, where production has expanded by over two-thirds since 1949-50.

Consumption of pyrites and other sulphurous materials amounted to 3,136,000 tons, or an increase of 50% over the tonnage used five years ago.

**Calcium carbide.** The figures submitted by seven of the twelve countries producing carbide, these seven countries producing over three-quarters of the total output of O.E.E.C. countries, indicate an increase in production of 13%.

The increase in production in France is largely due to the development of acetylene-based organic chemicals, in particular vinyl derivatives. This development probably explains why output has risen considerably over the last few years in other countries also; it would appear, however, that an increasing proportion of the acetylene used for this purpose is now obtained from oil or natural gas.

United States' sales of calcium carbide in 1954 amounted to 647,800 metric tons. Here, however, cracked petroleum hydrocarbons are increasingly used as raw material for the production of acetic acid and vinyl derivatives, and rising demand for these latter products is unlikely to make necessary any large increase in calcium carbide output.

**Primary ammonia.** The output of eight of the twelve countries producing primary ammonia shows an increase of 15% over 1953. Increased production in all countries was mainly due to the increased demand for nitrogenous fertilisers. The largest increases have taken place in Portugal where this industry has only recently been set up, and in Italy where substantial developments have taken place following the use of natural gas as a raw material.

**Imports and exports.** In 1954, inorganic chemicals accounted for about 17% of total imports and 15% of total exports of chemical products. Imports by Germany were over 100% higher in 1954 compared with 1953,

and imports by Austria, Denmark and the United Kingdom rose by over 40% also.

The increase in exports is shared relatively equally between the various exporting countries. The fall in dollar exports, probably due in part to a levelling-off in industrial activity in the U.S., should be noted.

In the United States, imports of inorganic chemicals accounted for 27% of total chemical imports in 1954, and exports for 9% of total chemical exports. For Canada the percentages were 12% for imports and 22% for exports. While Canadian imports and exports remained stable in 1954, compared with 1953, there was a fall of 15% in U.S. imports and a rise of 25% in exports compared with the previous year.

### Basic organic chemicals

Demand for methanol, particularly from the plastics industry, is rising rapidly and the output of five of the nine O.E.E.C. countries producing this chemical has nearly doubled since 1950.

Information for the U.K. is no longer available, since there is only one producer. United States' output in 1954 amounted to 510,400 metric tons.

Output of acetic acid seems to be rising more slowly than that of the other organic chemicals considered in the report, although the level of production in 1951 has now been greatly exceeded.

Acetone output seems to have increased in 1954, especially in France, where new processes based on cumene have been launched. United States' output fell by 8% compared with 1953, though the information on the first quarter of 1955 shows a marked improvement.

The effect of the installation of plant for the production of phenol by the cumene process is making itself felt in several countries and, with increased demand in particular for the manufacture of plastics materials, output has risen considerably.

With the development of plastics materials, the demand for phthalic acid compounds has risen considerably over the last few years. In the U.K., for instance, production of phthalate plasticisers increased by nearly two-thirds in 1954 compared with 1953, and output has been rising in Germany, Norway and Sweden.

O.E.E.C. countries' trade in organic chemicals amounted to 16% of total imports and 14% of total exports of chemicals in 1954.



## Prospects for basic chemicals

Throughout Western Europe the chemical industry is expanding and production of most basic chemicals is increasing, often very rapidly. Imports are, however, rising. The levelling off in demand in the United States may to some extent have stimulated exports in basic chemicals, but the rise in European imports from dollar sources would not seem to be primarily due to this. The reason will rather be found in the fact that sufficient supplies of basic chemicals have not always been available to meet the demands of manufacturers of semi-finished and finished products. Until European countries have increased their capacity for the production of a large number of basic chemicals, imports are likely to be maintained at a higher level than in the past, if Western Europe's chemical production is to continue to expand.

As stated in the report, the heavy investments to be carried out in this sector may be expected to remedy the situation in the not-too-distant future.

## Miscellaneous sectors

**Petroleum chemicals.** Total investment in O.E.E.C. countries' petroleum chemical plants rose from \$240 million at July 1, 1953, to \$290 million at December 31, 1954. Over this period, production rose very substantially. The main expansion took place in France and Italy rather than in the U.K., which had developed petroleum chemicals production on a substantial scale in the immediate post-war period.

The total expansion planned over the next 2½ years is very substantial, for the European petroleum chemical industry expects virtually to double its investments during this period.

In general it can be said that prices, in particular of petroleum chemicals sold in bulk, have shown a downward tendency over the past two years.

**Fertilisers.** The report draws attention to the fact that the O.E.E.C. Working Party on Nitrogenous Fertilisers recently considered plans for building nitrogenous fertiliser plants throughout the world. The information available shows such a high rate of increase in future production that it may be asked whether nitrogen producers in Europe and overseas will not find difficulty in marketing this large production in the fairly near future.

It is, of course, doubtful whether all the new plants will eventually be built. Though, in many cases, the new capacity will make use of cheap hydrogen supplies and is likely to

prove an attractive investment, lack of capital may prove one of the limiting factors in some underdeveloped countries. Shortage of skilled manpower may also create difficulties.

It seems probable that, even if not all the new plants are finally built (in general those scheduled to come into operation in 1956-57 are already under construction), there will be idle capacity in the near future unless some means can be found to stimulate demand for fertilisers in many parts of the world.

Production of phosphatic fertilisers increased from 1952-53 to 1953-54 for the first time for several years. The increase of 36% in production of concentrated superphosphates and, after a long period of stagnation, the increase of 10% in simple superphosphates are most worthy of note.

Production of potash fertilisers has increased and consumption also increased very considerably.

**Plastics materials.** The report gives, for the first time, comparable figures for sales of plastics materials in the main producer countries of Western Europe. Total sales more than doubled from 1950 to 1954, rising from 348,000 to 844,000 tons. This spectacular increase emphasises the fact that plastics materials are one of the sectors in which the chemical industry in Western Europe is expanding most rapidly. Four countries—Germany, the United Kingdom, France and Italy—accounted in 1954 for some 90% of total European sales.

Production of polyvinyl chloride is expected to continue its rapid expansion, and the new polythene plants now in course of planning or con-

struction will make a large and increasing contribution. Two interesting developments are a new method of polymerising ethylene (large plans are already under way in Germany for the production of this type of polythene) and new and improved types of polystyrene.

**Industrial explosives.** A high level of activity in the mining industry and expansion of capacity for the manufacture of hydroelectric power created a heavy demand for explosives, particularly in France and Norway. The Netherlands started to produce TNT for the first time.

## Sulphurous raw materials

The report points out that, at a time when production of sulphur as such is generally increasing in other parts of the world (production in the U.S., for instance, was higher by 7% in 1954 than in 1953), output in O.E.E.C. countries is tending to decline. The high cost price of Italian sulphur and the difficulties encountered on export markets resulting therefrom are the main reasons for this decrease, while the production of sulphur in France—never large, and only of interest to that country when supplies from other sources were very short—has now been suspended.

Recovery of sulphur, particularly recovery during the refining of oil, is being undertaken to an increasing extent in several countries and the quantity now recovered by O.E.E.C. countries from all sources is double that of 1948-49.

Production of sulphurous materials as a whole in O.E.E.C. countries now seems to be static. Consumption, however, was 12% higher in 1954 than in 1953. Imports in 1954 were nearly 50% greater than in the previous year, when they were, however, abnormally low. Compared with 1952, the increase is only 7%.

The supply situation seems, by and large, to be satisfactory at the moment, and European countries are getting all the sulphur they require. In general a market can be found for the sulphur produced in O.E.E.C. countries and only in Italy have difficulties been encountered.

It would appear that the increase in stocks in O.E.E.C. countries is partly due to the rise in sulphur stocks in Italy, since export sales are now negligible. It is, however, hoped that the measures under consideration by the Italian Government will permit the Italian producers to compete in future more satisfactorily on the world market.

## To Authors of Technical Articles and Books

*The Editor welcomes practical articles and notes on chemical engineering and related subjects with a view to publication. A preliminary synopsis outlining the subject should be sent to The Editor, CHEMICAL & PROCESS ENGINEERING, Stratford House, 9 Eden Street, London, N.W.1.*

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# PRESSURE DROP IN HEAT EXCHANGERS

By D. Venkateswarlu and P. M. Krishna

(Indian Institute of Technology, Kharagpur, India)

*The prediction of pressure drops of fluids flowing through heat exchangers is essential in the process design of exchangers so that safety factors need not be large. In exchangers which are to work under gravity flow of fluids or under vacuum, pressure drop determination is critical because the fluids can pass through the exchangers only if the resistance to fluid path is lower than the available head. A review of the available data and correlations for the determination of pressure drop in heat exchangers is presented in this article.*

THE principal parts of an exchanger fitted with a floating head, two passes on the tube side and one cross-baffle on the shell side are shown in Fig. 1. But exchangers may be fabricated in other ways also. The shell-side and tube-side fluids may flow in a single pass or in multipass. Multi-pass flows on the tube side and shell side are achieved by the provision of baffles in the distribution head and the shell, respectively. The shell-side baffles may be transverse or longitudinal. The commonly used transverse baffles, which cause the shell-side fluid to flow through the shell at right-angles to the axes of the tubes, are shown in Fig. 2. Impingement baffles, support plates and strip plates, if installed, offer additional resistance to fluid flow. The fluids flowing may be liquids or gases, condensing vapours or boiling liquids. The tubes themselves may be arranged in several ways as shown in Fig. 3, and the variations in arrangement affect the pattern of fluid flow and therefore the pressure drop. The flow paths of the different streams of the shell-side fluid in a cross-baffled exchanger are shown in Fig. 4. In view of the wide variations of possible constructions, physical properties of fluids and resulting varieties of fluid-flow patterns, a simple correlation for pressure drop through a heat exchanger is not possible, as in the case of flow through smooth pipes. As the flow patterns of the tube-side and shell-side fluids are altogether different, pressure drops of the tube-side and the shell-side fluids are considered separately.

## Tube-side pressure drop

The total pressure drop,  $\Delta P_t$ , of the fluid flowing through the tubes of

an exchanger is the sum of the pressure drops due to: (1) entrance to the header, (2) flow across the entrance header, (3) entrance to the tubes, (4) flow through the tubes, (5) exit from the tubes, (6) flow across the exit header and (7) exit out of the exchanger or for re-entry to the next pass.

Entrance and exit losses, items (3) and (5) above, are given by the following equations, respectively:

$$\Delta p_{ent} = \frac{kV_2^2}{2gc} \dots\dots\dots (1)$$

$$\Delta p_{exit} = \frac{V_1^2 - V_2^2}{2gc} = \frac{V_1^2}{2gc} \left(1 - \frac{A_1}{A_2}\right)^2 \dots\dots\dots (2)$$

Subscripts 1 and 2 refer to the inlet

and exit conditions;  $k$  is a function of  $D_1/D_2$ . Presuming that  $D_2/D_1 = 0$  for the entrance to the tubes and  $D_1/D_2 = 0$  for the exit from the tubes, the total loss under items (3) and (5) above becomes  $1\frac{1}{2}$  velocity heads per tube pass.

The pressure loss through the header and across the ends of the tubes to re-enter the tubes or to leave the exchanger cannot be easily estimated, but it is a safe practice to allow  $2\frac{1}{2}$  velocity heads for these losses.

The pressure loss which the fluid suffers when entering and leaving the exchanger may be allowed by providing  $1\frac{1}{2}$  velocity heads, one velocity head being equal to  $V_p^2/2gc$ , where  $V_p$  is the velocity in the pipe leading to and from the exchanger.

The pressure loss for flow inside the tubes is given by the well-known Fanning's equation:

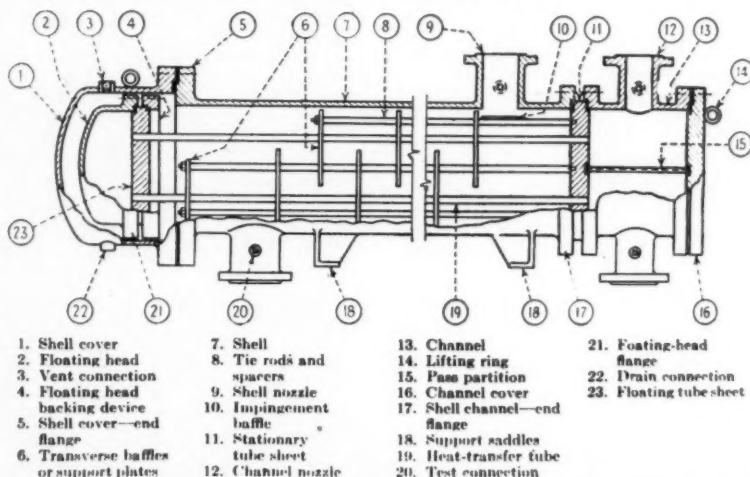


Fig. 1. Exchanger with one floating head, two tube passes and one cross-baffled shell pass.

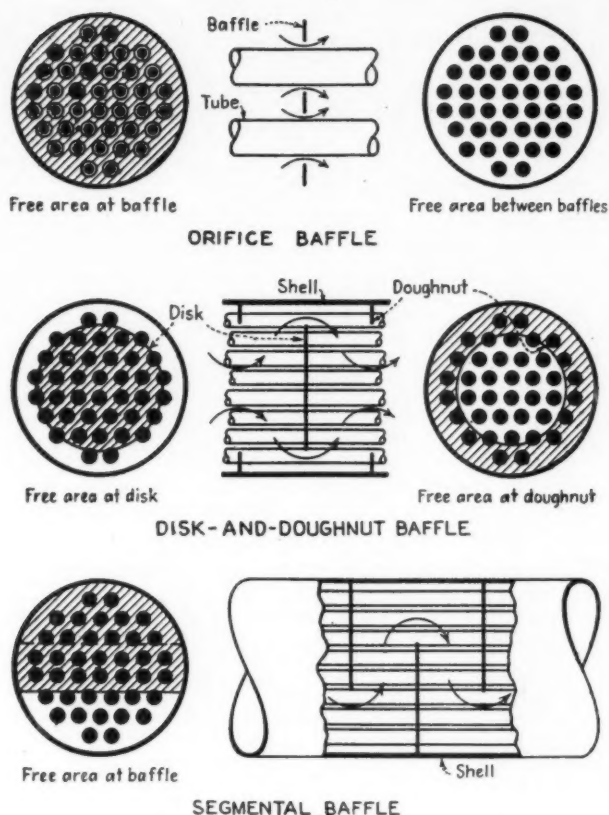


Fig. 2. Commonly used transverse baffles.

$$\Delta p_t = \frac{4f_t l \rho_i V_i^2}{2g_c D_i} \dots \dots \dots (3)$$

For a multipass exchanger with  $n$  tube passes, equation (3) may be written as:

$$\Delta p_t = \frac{4f_t l \rho_i V_i^2}{2g_c D_i} = \frac{4f_t l G_i^2}{2g_c \rho_i D_i} \dots (4)$$

The friction factor  $f_t$  may be read from friction factor *versus* Reynolds number plots or calculated from equations (5) and (6) for viscous and turbulent flows, respectively.

$$f_t = \frac{16}{D_i G_i / \mu_i} \dots \dots \dots (5)$$

$$f_t = 0.00140 + \frac{0.125}{(D_i G_i / \mu_i)^{0.32}} \dots (6)$$

In the commercially important region of flow (*i.e.* Reynolds range of 5,000 to 1,000,000), the value of  $f_t$  ranges from 0.004 to 0.01 for clean commercial pipe. A value of 0.0055 is a good average for the entire range. Thus the total pressure drop for the tube-side fluid of an exchanger is given by:

$$\Delta P_t = \frac{4f_t l G_i^2}{2g_c \rho_i D_i} + \frac{1.5 V_p^2}{2g_c} + \frac{4 V_i^2 n}{2g_c} \quad (7)$$

The value of the last term in equation (7) is generally small in comparison with the other terms and hence this term may be neglected.

#### Shell-side pressure drop in unbaffled exchangers

The pressure drop of the fluid flowing on the shell side of an unbaffled exchanger is the sum of the pressure drops due to:

- (1) Enlargement and contraction in flowing through the nozzles.
- (2) Flow past the tubes, which may be assumed to be parallel to the axis of the shell. This assumption is correct only when the tube length is large compared to the tube diameter.

Pressure drop due to enlargement and contraction in entering and leaving the shell may be estimated by equations (1) and (2) given above.

Pressure drop of fluid flowing parallel to tubes may be estimated from the equation:

$$\Delta p_o = \frac{4f_o V_o^2 \rho_o l}{2g_c D_e} \dots \dots \dots (9)$$

The equivalent diameter  $D_e$  is taken as four times the mean hydraulic radius (total flow area/total wetted perimeter). Shell-side fluid velocity  $V_o$  is calculated on the basis of the total free cross-sectional area not occupied by the tubes. The friction factor  $f_o$  may be taken from the plot of friction factor *versus* Reynolds number for flow inside pipes, Reynolds number being defined as  $D_e V_o \rho_o / \mu_o$ .

The pressure losses which occur in unbaffled exchangers are very small compared to those which occur in baffled exchangers.

#### Shell-side pressure drop in baffled exchangers

The pressure drop of the fluid flowing on the shell side of a baffled exchanger depends on the flow pattern of the fluid. This in turn depends upon the following factors:

- (1) *Tube bank arrangement.* Tube spacing (transverse or longitudinal), tube-alignment characteristics (in line or staggered) and number of rows of tubes.
- (2) *Baffles.* Shape, size and spacing of baffles.
- (3) *Operating conditions.* Fluid flow rate and fluid properties.

In addition to these factors, a number of other factors such as tube roughness and wall frictional effect will affect the pressure drop, though to a smaller extent.

The pressure drop of the fluid flowing on the shell side of a baffled exchanger is the sum of the pressure drops due to: (1) enlargement and contraction losses in flowing through the nozzles; (2) flow across the tube bundle; and (3) flow through the baffle openings. The first of these is already considered. The pressure drop due to flow across tube bundle and through the baffle openings is considered below.

#### Flow across tube bundle

The determination of the pressure drop across the tube bundle is complicated because of the uncertain but appreciable flow reduction due to leakage through the clearances between tubes and the baffles and between the shell and the baffles as shown in Fig. 4. Also, the pattern of flow is indefinite, since a part of the fluid may flow for some distance parallel to the tubes following a change of flow direction, particularly if the baffle spacing is high. Further, the shell being cylindrical, the number of tubes per row and therefore the free cross-sectional area and mass velocity vary at the different levels of



the tube bundle. Lastly, the tube spacing and arrangement vary considerably in the different units and any equation for pressure drop must make allowance for these variations, which affect the pressure drop considerably.

There is a large amount of experimental data reported on the pressure drop of fluids flowing across tube bundles. In 1948, Boucher and Lapple<sup>1</sup> gave complete references, details of tube sizes and tube arrangements, conditions of flow, range of studies involved and limitations of all the reported data that were available at that time. In 1950, Bergelin *et al.*<sup>2</sup> reported pressure-drop data during viscous flow across unbaffled tube banks obtained by working on seven ideal heat-exchanger models in which the effects of leakage, by-passing and flow in directions other than normal to the tubes were eliminated or minimised. The work was later extended<sup>3</sup> to cover the transition zone between the viscous and turbulent flow regions. Later still, another paper<sup>4</sup> reported fluid friction data during flow across banks of tubes working with a cylindrical exchanger containing baffles, which is equivalent to a commercial shell-and-tube exchanger in which some modifications have been made to simplify the internal flow pattern.

Based upon their own experimental data or the data of others available to them, a number of investigators presented correlations for pressure drop. Some correlations, like those of Andreas,<sup>5</sup> are limited in the range of spacings and configurations investigated by these authors. There are some other reported correlations, such as those of Berlich and Thein,<sup>6</sup> which have been shown by Boucher and Lapple<sup>1</sup> to be either 'incomplete, incorrect or difficult to use.'

The correlations generally found useful are those of Chilton and Genereaux,<sup>7</sup> Grimson<sup>8</sup> and Gunter and Shaw.<sup>9</sup> The correlation proposed by Chilton and Genereaux is based upon modified Reynolds number,  $Re_{mc}$ , and friction factor,  $f_{ct}$ , which are defined by:

$$Re_{mc} = \frac{D_c G_{max}}{\mu_o} \dots \dots (10)$$

$$f_{ct} = \frac{\Delta P_o}{4N} \cdot \frac{G_{max}^2}{2\rho_o g_c} \dots \dots (11)$$

The clearance between tubes,  $D_c$ , corresponding to minimum area of flow section, makes a partial allowance for the effect of transverse spacing. Longitudinal spacing is known to have only a minor effect for staggered arrangements. The friction factor,  $f_{ct}$ ,

is based on  $N$ , the number of major restrictions encountered in flow through the bank. The value of  $N$  is taken as the number of rows of tubes normal to flow for staggered tubes with minimum area in transverse openings and for in-line tubes. But, in the case of staggered tubes with minimum area in the diagonal openings,  $N$  is taken as the number of tubes less one.

Using Chilton-Genereaux correlation, the pressure drop for the shell-side fluid flowing through a shell of circular cross-section is given by:

$$\Delta P_o = \frac{4f_{ct} N_r \rho_o V_{max}^2}{2g_c} \dots \dots (12)$$

The value of friction factor,  $f_{ct}$ , is given by:

$$f_{ct} = 0.75 \left( \frac{D_c G_{max}}{\mu_o} \right)^{-0.2} \dots \dots (13)$$

which is recommended for commercial spacings with transverse pitches in the range of 1.25 to 1.50 tube diameters. Average values of number of tube clearances per row and of tube clearance,  $D_c$ , must be used in order to allow for the variations in the number of tubes per row and in the clearance spaces in shells of circular cross-section. For multipass exchangers,  $N_r$  is the number of rows of tubes in the whole exchanger, which is the number of tubes in a pass multiplied by the number of passes.

Grimson<sup>8</sup> correlated the data of Hoge<sup>10</sup> and Pierson<sup>11</sup> and presented a series of charts. These charts give the friction factor,  $f_{ct}$ , as functions of modified Reynolds number,  $Re_{mt}$ , for staggered and in-line arrangements with longitudinal and transverse spacings as parameters for the modified Reynolds range of 2,000 to 40,000. Modified Reynolds number,  $Re_{mt}$ , is based on the outside diameter of the tubes. Jakob<sup>12</sup> presented the following equations correlating the friction factor data of Grimson's charts:

$$f_{ct} = \left( 0.44 + \frac{0.08x_L}{(x_T - 1)^{m_1}} \right) \left( \frac{D_o G_{max}}{\mu_f} \right)^{-0.15} \text{ for tubes in line} \quad (14)$$

$$f_{ct} = \left( 0.023 + \frac{0.11}{(x_T - 1)^{1.08}} \right) \left( \frac{D_o G_{max}}{\mu_f} \right)^{-0.15} \text{ for staggered arrangement} \dots \dots (15)$$

$m_1$ , in equation (14) is given by:

$$m_1 = 0.43 + \frac{1.13}{x_L} \dots \dots (16)$$

Jakob's equations are found to give satisfactory values in the  $x_T$  range of 1.5 to 4.

The correlation of Gunter and Shaw<sup>9</sup> is based upon modified Reynolds number and friction factor

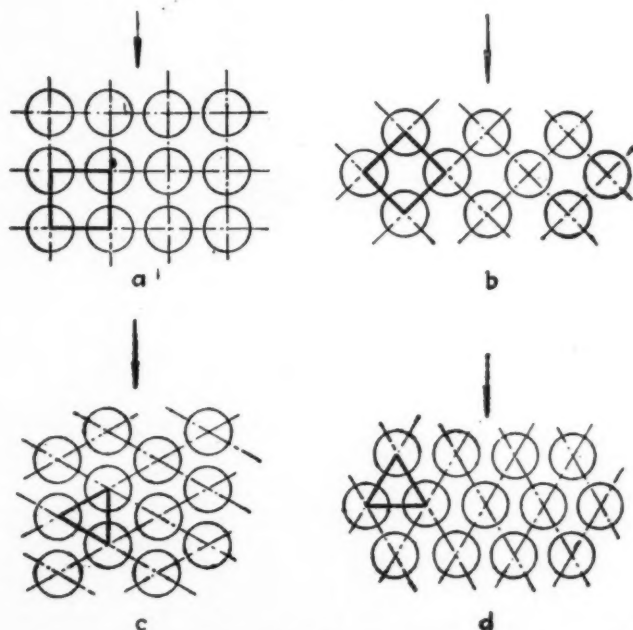


Fig. 3. Arrangements of tubes: (a) In-line arrangement with square pitch; (b) Staggered arrangement with square pitch; (c) and (d) staggered arrangements with triangular pitch.

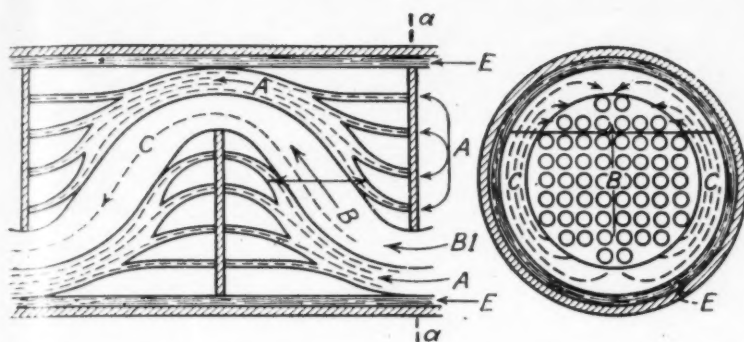


Fig. 4. Flow paths of shell-side fluid in a cross-baffled exchanger: (A) Flow through clearances between baffles and tubes; (B.) flow transversely across the tubes; (C) flow circumferentially around the bundle to the next position; (E) longitudinal flow through clearance between the baffles and the shell.

defined by the following equations:

$$Re_{me} = \frac{D_e G_{max}}{\mu_o} \quad (17)$$

$$f_m = \frac{\Delta P_o D_e}{L} \left( \frac{\mu_o}{\mu_w} \right)^{0.14} \left( \frac{s_T}{D_e} \right) \left( \frac{s_L}{s_T} \right) \frac{G_{max}^2}{2 \rho_o g_c} \quad (18)$$

In defining  $Re_{me}$ , volumetric hydraulic diameter is taken as the characteristic length. The modified friction factor makes allowance for variations in tube spacings. By thus defining Reynolds number and friction factor, Gunter and Shaw obtained a single curve for both in-line and staggered arrangements and for viscous and turbulent flows.

Boucher and Lapple<sup>1</sup> made a critical study of the correlations of Chilton and Genereaux, Gunter and Shaw, and Grimson. They concluded that the graphical correlation of Grimson is the best available for both in-line and staggered arrangements over the Reynolds range 2,000 to 40,000.

Bergelin *et al.*<sup>4</sup> working with an experimental baffled exchanger without internal leakage, found that, for turbulent flow, the pressure drop per restriction of a crossflow section is about the same as that of a simple crossflow exchanger of similar tube arrangement, but without baffles. When the same pressure-drop data is plotted as friction factor *versus* modified Reynolds number based on the outside diameter of tube, some spreading is observed in the turbulent region, due to baffle configuration. In the viscous flow region, the friction factors are lower than those for turbulent flow for the same cross-flow exchanger.

Tinker<sup>13</sup> developed a method for

evaluating the effective area of an exchanger from the exchanger dimensions, but his attempt at developing a correlation between friction factor and effective Reynolds number (using effective mass velocity based on effective area) has not been very successful.

In the transition region between the viscous and turbulent flows, Bergelin *et al.*<sup>3</sup> determined the pressure drop across banks of tubes in unbaffled experimental units and plotted friction factor *versus* modified Reynolds number. Their data for staggered arrangements are represented by curves which are without irregularities, but, for in-line arrangements, the curves show dips similar to those known for flow inside pipes. This difference in behaviour between staggered and in-line arrangements is due to a difference in the mechanism of turbulence formation.

In the viscous region, Bergelin *et al.*<sup>2</sup> worked on several heat-exchanger models in which the effects of leakage, by-passing and flow in directions other than normal to the tubes are eliminated or minimised. They correlated their experimental data by the equation:

$$f_{ct} \left( \frac{s}{D_o} \right)^{1.6} \left( \frac{\mu_o}{\mu_w} \right)^{0.57} Re_{me}^{-0.25} = \frac{70}{Re_{me}} \quad (19)$$

$$\text{where } Re_{me} = \frac{D_e G_{max}}{\mu_o} \quad (20)$$

$$\text{and } f_{ct} = \frac{2 \Delta P g_c \rho_o}{4 G_{max}^2 N} \quad (21)$$

#### Flow through baffle openings

Baffles which are fitted to heat exchangers in order to increase the shell-side heat-transfer coefficient by increasing the mass velocity may be of several shapes. Shapes of baffles which are commonly employed are

shown in Fig. 2. Orifice baffles which are generally used for moderate flow rates and non-fouling liquids force the liquid at high velocity through the clearances between the baffles and the tubes. In the disc-and-doughnut baffles, the disc baffle has an annular flow area between the baffle and the shell, while the doughnut baffle has a central opening for fluid flow. The discs and doughnuts are placed alternately with equal distances in between. These baffles, therefore, direct the fluid to flow alternately from the shell side towards the centre of the tubes and back again towards the shell side. The segmental baffles are formed by cutting out a segment along a horizontal chord from a circular metal plate of slightly smaller diameter than the inside diameter of the shell. The size of the opening may be 15 to 45% of the shell area. It may be readily seen from Fig. 2, that the pattern of fluid flow is considerably altered by the baffle shape.

The flow pattern is also modified by the clearances between the baffle and the shell, the tubes and the baffles, and the tube bundle and the shell. These clearances are provided to facilitate withdrawal of tubes or tube bundles for cleaning or replacement. At the same time, care is taken to provide only the minimum required clearance so that by-passing of fluid is minimised and high heat-transfer coefficient is ensured. Thus a clearance of  $\frac{1}{8}$  to  $\frac{1}{4}$  in. is usually left between the i.d. of shell and o.d. of baffles, and tube holes in the baffle plates are drilled about  $\frac{1}{32}$  in. larger than the o.d. of tubes.

Another factor which makes the fluid-flow pattern uncertain is the shell space which is not fitted with tubes. Though uniform distribution of tubes in the shell is essential, structural considerations necessitate leaving an annular space between the tube bundle and the shell vacant. Lanes are also left in the tube layout so that partition plates may be fitted.

Huge<sup>10</sup> gives the following equation for roughly estimating the pressure drop across a segmental baffle opening in turbulent flow:

$$\Delta P_B = \frac{2 \rho_o V_B^2}{2 g_c} \quad (22)$$

Actually, the pressure loss across a given opening will be markedly affected by the baffle spacing and whether or not the tubes pass through the opening. Because of leakage, the above equation is considered conservative and further refinements are hardly justified.

Donohue<sup>14</sup> analysed the experimental test figures of Short<sup>15</sup> for turbulent flow of water through segmental baffle openings and correlated the pressure drop through baffle openings by the equation:

$$\Delta P_B = 2.005 V_B^2 \rho_o \dots (23)$$

Bergelin *et al.*<sup>4</sup> studied the pressure drop in an experimental baffled exchanger in which internal leakage is eliminated. Pressure drop across the baffle opening is taken as due to flow of fluid past the tubes and due to reversal of direction. Taking the pressure drop due to reversal of direction as equivalent to two velocity heads, the total baffle opening pressure drop for turbulent flow is given by the equation:

$$\Delta P_B = (2 + 0.6N_B) \frac{\rho_o V_z^2}{2g_c} \dots (24)$$

where  $N_B$  is the estimated average number of restrictions in baffle opening and  $V_z$  is the geometric mean of the velocities through the average of minimum cross-sectional areas for rows of tubes in cross-flow section and through the free cross-sectional area of baffle window. But equation (24) is applicable for exchangers in which clearances are absent and therefore full penetration prevails and is not applicable to industrial heat exchangers such as the floating head, removable tube, bundle-type exchangers. The same authors intend to get the data for heat exchangers with finite but controlled leakage. In the absence of such data, it is safe to use equation (23) for determining the pressure drop through baffle opening.

This review indicates that the determination of pressure drop of the shell-side fluid of a commercial heat exchanger is not simple, due to the large number of variables which affect the pattern of fluid flow. Though the trend in design is to minimise the leakages by reducing the clearances wherever possible, the flow pattern still remains uncertain. Basic research is needed to get a clear concept of the effect of baffles and by-pass routes on flow pattern. Also, analysis of performance of commercial exchangers whose specifications and flow conditions are completely known, are needed. Work on these lines is being carried out by Bergelin and co-workers of the University of Delaware.<sup>2-4</sup> The study of the mechanism of fluid flow under pure cross-flow conditions and of the effect of baffles and clearances is being undertaken at the Institute of Technology, Kharagpur.

## NOMENCLATURE

- $A$  = Area of cross-section;  $A_1$  at the entrance;  $A_2$  at the exit.  
 $D$  = Diameter;  $D_1$  at the entrance;  $D_2$  at the exit;  $D_i$  inside diameter of tubes;  $D_e$  equivalent diameter;  $D_c$  clearance between tubes corresponding to minimum area through which fluid flows.  
 $f$  = Friction factor;  $f_i$  for flow inside tubes;  $f_o$  for flow outside and parallel to tubes;  $f_{ct}$  friction factor for flow in cross-baffled exchangers defined by  $(\Delta P/4N) (G_{max}/2\rho_o g_c)$ ;  $f_m$  friction factor defined by Gunter and Shaw<sup>9</sup> making allowance for tube spacing, equal to:  $(\Delta P_o D_e/L) ((\mu/\mu_w)^{0.14} (s_T/D_e) (s_T/s_L)^{0.8}) / (G_{max}^2/2\rho_o g_c)$ .  
 $g_c$  = Gravitational constant.  
 $G$  = Mass velocity;  $G_i$  for fluid flowing inside tubes;  $G_{max}$  maximum velocity of shell-side fluid based on minimum available flow cross-sectional area.  
 $k$  = Constant.  
 $l$  = Length of tubes.  
 $n$  = Number of tube side passes;  $N$  number of major restrictions encountered in flow through the bank;  $N_r$  number of rows of tubes in the whole exchanger;  $N_B$  average number of restrictions in baffle opening.  
 $\Delta P_i$  = Total pressure drop of tube side fluid;  $\Delta p_{ent}$  due to entrance to the header;  $\Delta p_{exit}$  due to exit from the tubes;  $\Delta p_o$  total pressure drop due to flow outside and parallel to tubes;  $\Delta P_o$  due to cross-flow of shell-side fluid;  $\Delta p_B$  pressure drop through baffle opening;  $\Delta p_i$  pressure drop due to flow inside the tubes.  
 $Re$  = Reynolds number;  $Re_{mc}$  modified Reynolds number defined by  $D_e G_{max}/\mu_o$ ;  $Re_{mt}$  defined by  $D_o G_{max}/\mu_o$ ;  $Re_{me}$  defined by  $D_o G_o/\mu_o$ .  
 $s$  = Tube pitch;  $s_T$  transverse pitch;  $s_L$  longitudinal pitch;  $s_{L'}$  longitudinal pitch as defined by Gunter and Shaw.<sup>9</sup>  
 $V$  = Velocity of fluid;  $V_B$  of fluid through baffle opening;  $V_i$  of fluid flowing inside the tubes;  $V_o$  of fluid flowing outside the tubes;  $V_{max}$  maximum velocity based on minimum available flow cross-sectional area;  $V_p$  of fluid in the pipe leading to or

from the exchanger;  $V_z$  geometric mean of the velocities through the average of minimum cross-sectional areas for rows of tubes in cross-flow section and through the free cross-sectional area of baffle window.

- $x_L$  = Ratio of longitudinal pitch to outside diameter of tubes;  $x_T$  ratio of transverse pitch to outside diameter of tubes.  
 $\rho$  = Density of fluid;  $\rho_i$  of tube-side fluid;  $\rho_o$  of shell-side fluid.  
 $\mu$  = Viscosity of fluid;  $\mu_i$  of fluid flowing inside the tubes;  $\mu_o$  of fluid flowing outside the tubes;  $\mu_f$  fluid at the film temperature;  $\mu_w$  of fluid at the mean mass temperature.

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**Carbonitriding.** This process of case-hardening steel, discussed in an illustrated pamphlet from Wild-Barfield Electric Furnaces Ltd., involves the simultaneous introduction of the elements carbon and nitrogen from a suitable atmosphere. The atmosphere can either be prepared in a separate unit and fed to the furnace, or it can be produced *in situ* by the drip feed method. According to the pamphlet, carbonitriding may be carried out very simply by the Wild-Barfield process, which is based on the use of raw town's gas and ammonia. Various types of equipment are described.



# Separating Fluids by Gas-Liquid Partition Chroma- tography

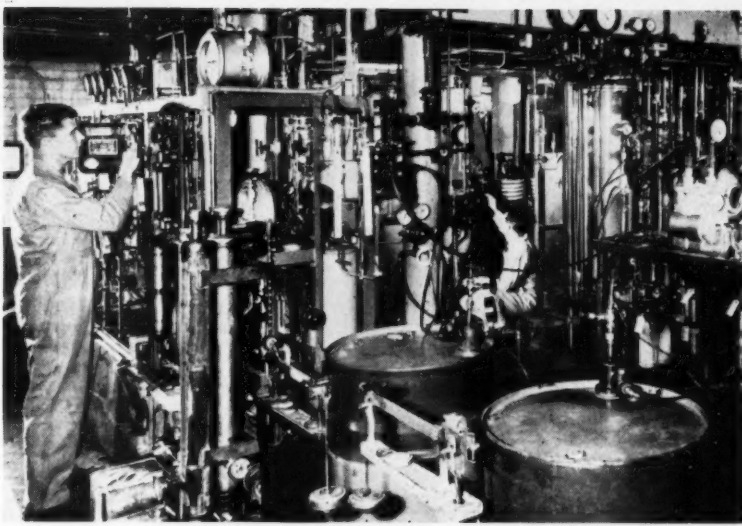
*American application  
of British discovery*

RESEARCH scientists of the American Gulf Oil Corporation have recently found a way to 'out-distill' the most high-powered, modern research stills, using the principles of gas-liquid partition chromatography. Their new technique may bring untold benefits to the users of chemical and petroleum products.

The method can be used for quickly separating gases and liquids whose



Operating engineer injects minute chemical sample into gas-liquid partition chromatography unit. After the sample has been injected, the unit operates automatically and the recorder plots the concentrations of the components in the sample.



The work of petroleum processing pilot plants, like one shown in the picture, will be benefited by the more comprehensive separation of components made possible by gas-liquid partition chromatography. The speed, economy and scope of the technique developed at the American Gulf Oil laboratories will help both to control pilot plant operations and to study the products and efficiency of the processes under investigation.

boiling points are so close that the most elaborate present-day distilling columns either cannot separate them at all, or require weeks to do so. It also separates materials in quantities too small for distillation, permitting analysis where only traces are available and conserving costly research materials. In fact, a hypodermic needle is used to feed in the samples.

The apparatus used is a so-called chromatographic tube, this being simply a long, thin tube filled with a granular powder which laboratory technicians coat with an oily substance. The tube is maintained at a constant temperature in a range up to 300°F.

When a fluid to be studied is injected, part of it goes into solution with the oily matter on the grains and the remainder becomes a gas in the spaces between the grains. A 'carrier gas' is next introduced to force the gas in the spaces to move forward. Additional matter then comes out of solution into the voids between the grains, and is in turn moved forward by the carrier gas.

This process goes on continuously while the flow of carrier gas is maintained. It results in different components of the material under study moving down the tube at different rates, because each displays a different equilibrium between its soluble phase in the oily material and its gaseous phase in the spaces between grains.

As each component emerges from the end of the tube, it is detected by

a conventional thermal conductivity cell and identified on the basis of its transit time through the tube.

## More exact analysis

The key to this new technique is the fact that, under the conditions described, the various compounds in a mixture can be made to move forward in a tube at different speeds and so can be easily separated at the end. Its significance lies in opening the door to the more exact knowledge of materials needed for producing higher-efficiency oils, petroils, chemicals and plastics in the future. Further progress in these fields requires analysis of compounds to increasingly finer degrees.

Gas-liquid partition chromatography is based on a phenomenon in physical chemistry which has only recently been introduced to practical use by British scientists. Learning of the British work in 1954, Gulf scientists instituted intensive research for applying it to the analysis of industrial petroleum and chemical materials. Successful results were reported to the scientific world at the 1955 Pittsburgh conference on analytical chemistry and applied spectroscopy.

The new technique has been described as one of the most basic advances in separating fluids since man learned to boil off alcohol. It has also been said that the new type of chromatography is to the distillation column what the column was to the primitive still.

# Automatic Pressure Butt Welding of High Pressure Pipe Lines

NEW PROCESS FOR USE WITH HIGHLY STRESSED COMPONENTS

THE Burton pressure butt-welding process was initially developed to overcome production bottlenecks in quality pipe welding and employs the novel principle of allowing the plasticity of the steel being welded to control its own welding cycle. Development work on this process was commenced in 1947 at H.M. Dockyard, Rosyth, and later at the Central Metallurgical Laboratory, Emsworth, and H.M. Dockyard, Portsmouth.

Essentially the process consists of bringing the faces to be joined together under a minor load and heating by means of a multi-jet oxyacetylene oscillating ring burner until a pre-set thermal expansion is reached. A second higher or control load is then applied to return the pipe to its original length, when the final butting pressure operates to complete the weld. All sequences are controlled automatically by limit switches which are also arranged to control upset and to cut off the gas supply just before completion of the weld.

The system of control is shown schematically in the accompanying diagram. It will be noticed that the welding operation is controlled by a rod, carrying the limit switches, locked to the moving part of the machine, which by recording linear dimensional changes causes the welding sequence to occur. Welding temperatures are not measured, but may be systematically varied by alteration of the control pressure; raising the pressure reduces and lowering increases weld temperature. Working pressure limits for the production of satisfactory welds are generous and welding times lie between 45 sec. and 4 min. according to pipe wall thickness.

## Equipment and applications

Two practical machines using these principles have been constructed, one for shop use and the other a prototype portable model. The development of the portable machine was undertaken in view of the particular advantages foreseen in the application of this process to the welding of pipes on site and in positions of limited access. The 'shop' model can be adapted as a transportable machine.

The Portsmouth 'shop' machine, by the use of different-size ring bur-

ners and collet holders, has been constructed to weld pipe to pipe and pipe to flange in sizes from 3 to 8 in. o.d. for wall thicknesses between  $\frac{3}{16}$  and  $\frac{3}{4}$  in. Provision has also been made for tube turn to pipe and tube turn to flange welds.

The portable machine, for pipes 1½ to 2½ in. o.d. and ¼-in. wall thickness, consists of three separate units: the welding head, the hydraulic cabinet and the connecting leads. The design is such that the welding head may be assembled round the pipe in any position.

In both machines an oxyacetylene flame on the reducing side of neutral is used and commercial flowmeters and a back-flash arrester are fitted in the gas supply, which is by manifolded

cylinders of oxygen and acetylene. Burn-back arresters are fitted direct to each half of the ring burners. Joint preparation consists of a fine-machined bevel finish with mating faces on the bore of the pipe. Weld upset is controlled to be between 50 and 60% and no subsequent machining of inside or outside reinforcement is required, as significant edge weaknesses are absent.

Welding has been confined to mild, carbon-molybdenum and chromium molybdenum steel pipes, but there does not seem to be any reason why more highly alloyed pipe steels cannot be joined by this process. Post-weld heat treatment is a requirement and consists of normalising for mild-steel pipes and normalising and tempering for low-alloy steels where air-harden-

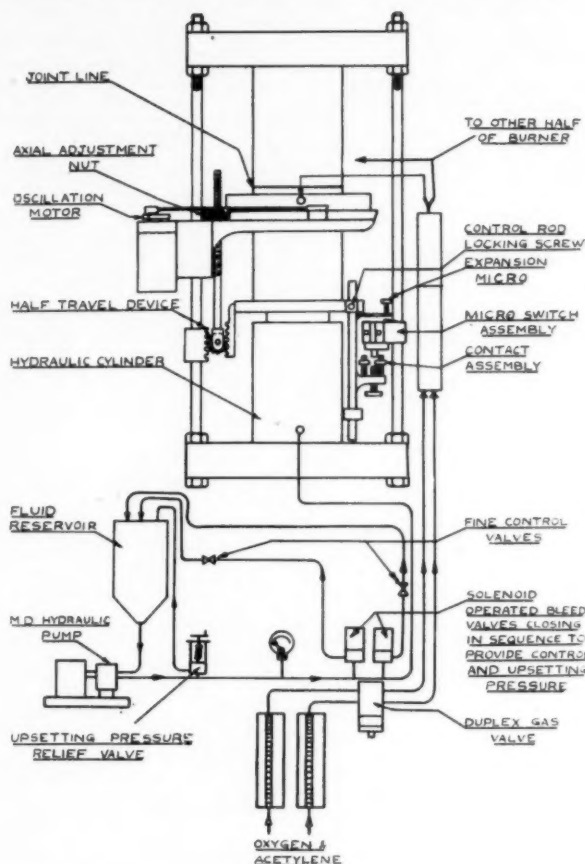


Diagram showing system of control of pressure butt welding machine.

ing tendencies are present. Straight normalising may be carried out on the machine, but tempering heat treatments require additional equipment.

Bend tests with either the inside or outside reinforcement removed have been used for test purposes during development. Such tests have proved to be most discriminative and readily capable of detecting inside or outside edge weakness at the weld line. For production control the removal of a small amount of reinforcement in the form of a chip  $\frac{1}{16}$ -in. thick is practised; a surface defect will cause the chip to part at the weld line.

Micrographic examination of the inside surface of a good mild-steel weld shows complete absence of faults.

The weld line may be shown to be richer in oxygen than the adjacent parent metal. Such zones have similar properties to the parent pipe and are regarded as acceptable, provided surface metallurgical notches are absent.

#### Applications other than pipe welds

Apart from pipe welds the possibility of using this process for other highly stressed components such as gas cylinders and small pressure vessels should not be overlooked. It should be emphasised that the process is fully automatic and, apart from the necessity of ensuring correct setting of the machine and occasional consistency checks, highly skilled operators

will not be required. Accordingly, time saving over manual or automatic metal arc welding may be expected to lead to considerable economies.

The Burton process is covered by British Patent No. 706406 and the patent is owned by the National Research Development Corporation. The development of a workshop automatic butt-welding machine from the commercial aspect has been entrusted to Metropolitan Vickers Electrical Co. Ltd., under contract to the Admiralty.

The patent development was originated by Mr. J. E. Burton, a dockyard technical officer, who has been appointed as technical adviser on the development of the process on the machines that now bear his name.

## Explosion Reliefs for Drying Ovens

*About 40% of explosions in gas-heated plant in Britain occur in drying ovens, and experimental work on this problem has been started by the Gas Council. Important new conclusions that have been reached so far will not only bring greater safety in the use of box ovens, but will also shed light on various other types of explosions in industry. The data obtained have been set out in a paper\* presented to the Institution of Gas Engineers by P. A. Cabbage, B.S.C., and W. A. Simmonds, PH.D., F.INST.P., and which deals with the design of top reliefs for box ovens. Here are some extracts from this paper.*

THE casualties and damage caused by explosions in the ovens commonly used in industrial drying processes have for some time caused concern to the Factory Department of the Ministry of Labour and National Service. These explosions are due to combustion of either the vapours formed in the drying process or the fuel used to heat the ovens. Since the large majority of these ovens are gas-operated, an investigation aimed at preventing the damage caused by these explosions was undertaken by the Gas Council.

Owing to the difficulty of eliminating explosions under industrial working conditions, attention has been confined to the prevention of damage by the use of explosion reliefs. Since many more of the explosions occur in box ovens than in conveyor ovens, the first objective was to obtain design data to enable safe explosion reliefs to be inserted in box ovens. As the usual position of a relief is in the top of an oven, data for top reliefs were obtained first, and are presented in this paper.

The steps involved in designing an

explosion relief for insertion in the top of a box oven are, firstly, the construction of the relief in such a way that it is harmless in operation; secondly, the calculation of the necessary vent area and greatest allowable weight of the relief to prevent damage to the oven; and, thirdly, the determination of the free space necessary above and around an oven to avoid interference with the operation of the relief and to eliminate the danger of burns.

Methods of dealing with each of these three steps are given for ovens that are approximately cubical and have neither solid shelves nor shelves that are virtually solid through heavy loading with stock. Some points that emerge in a consideration of these three steps are as follows.

#### Constructing a safe type of relief

In designing explosion reliefs for box ovens, it is just as important to prevent the relief becoming a dangerous missile when an explosion occurs as to prevent parts of the oven

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being hurled about. Such explosion reliefs as have been examined, in addition to being much too heavy, often suffer from the defects of not providing a seal and being easily prone to jam in the vent. When this happens, the oven effectively has no explosion relief.

A form of explosion relief was therefore evolved that would withstand industrial conditions and yet not form a dangerous missile. This was based on the considerations that the relief would break upon being projected from the oven, decelerate rapidly in the air, not rebound violently on impact with solid surfaces, and cause no injury on hitting anybody. This relief was supported on a base of expanded metal, which was built into the oven and consisted of a layer of aluminium foil, to provide a seal, covered with a layer of heat insulation. The type of expanded metal was chosen so as to support someone stepping on it accidentally and at the same time cause as little blockage of the vent area as possible.

These reliefs may be fitted into single- or double-cased ovens without any difficulty. Treble-cased ovens present an added complication, but if the heating surface over the top of the inner case can be dispensed with, an exactly similar type of relief may be used for treble- as for single- and double-cased ovens. If necessary, the spaces through which the products of combustion flow could be vented by reliefs at the top of the sides and back. If the heating surface in the top of the oven is essential to the oven performance, the type of vent described



above may be used in the top of the oven as a relief to atmosphere, but it is also necessary to insert a relief in the top of the inner case of the oven. The practice, which is quite common, of using a light, dished metal plate resting in an aperture in the top of the inner case cannot be condemned too strongly. This method makes nonsense of the principle of the treble-cased oven, since there is no seal between the products of combustion and the working space of the oven, and may even cause explosions.

Procedures are given for determining the weight and area of a relief when the strength of the oven is known, and when the strength of the oven is not known.

#### Clearance around oven

When using an explosion relief in the top of an oven, it is necessary to allow a clear space over the oven so that the relief can be flung clear of the vent. The clearance must be sufficient

to ensure that, if the relief rebounds, it will not fall back on to the oven in time to interfere with the venting. It must also be sufficient to ensure that there is an adequate venting area in a vertical direction between the edges of the relief and the top of the oven.

It is also necessary to ensure that, when the oven is in operation, nobody is in the region filled with the flame vented from the oven. This region can be determined from the measurements of the flames observed for town gas explosions. The height of the flame depends on the vent area coefficient and the oven volume. For a relief of the type recommended in the paper (which covers the whole of the top of the oven) this height is 4 ft. for a 100-cu.ft. oven and is given by

$$4\left(\frac{V}{100}\right)^{\frac{1}{3}} \text{ ft.}$$

(where  $V$  is the oven volume in cubic feet) for other oven sizes.

## Electrolytic Manganese from Low-grade Ores

IT has been the practice in the Indian manganese mining industry to select only high-grade ores (usually 45% Mn and above) for export, with the result that low-grade ores, though industrially useful, are accumulating in hundreds of thousands of tons as rejects at the mining centres in Madhya Pradesh, Orissa and Andhra.

Beneficiation of the low-grade ores, with a view to upgrading them, by washing, froth flotation or other operations is one of the solutions suggested; another is the production of electrolytic manganese metal of high purity.

The electrolytic production of manganese is discussed by A. Jogarao, Y. D. Prasada Rao and S. Gopal, of the Central Electrochemical Research Institute, Karaikudi, in a paper which appeared in the *Journal of Scientific and Industrial Research*, 1955, **14B** (9), 467-472. They point out that the electro-winning of manganese, unlike that of zinc and copper, demands a more delicate control which presents particular difficulties, more especially when low-grade ores are dealt with. It was, therefore, considered desirable to set up a semi-pilot plant and to carry out trials with indigenous low-grade ores. A daily output of 10 lb. of manganese metal of over 99.9% purity has been achieved.

The essential steps of the process are: (1) conditioning of the ore for

leaching; (2) leaching the conditioned ore with anolyte overflow from the electrolytic cells; (3) purification of the pregnant leach from deleterious impurities; (4) electrolysis of the purified solution; and (5) stripping of the manganese metal from the stainless-steel cathode sheets.

For conditioning, about 40 to 45 lb. of the ore were ground to 100 mesh, mixed with requisite quantities of charcoal and reduced in three batches of roasting at 600 to 700°C. in cast-iron crucibles. The calcine was cooled overnight, out of contact with air.

The leaching of the calcined ore was carried out for about 2 hr. in two lead-lined wooden vats containing anolyte overflow and sulphuric acid until the leach liquor attained the required manganese concentration of about 32 to 36 g./l.

The leach liquor was neutralised with ammonia and, after stirring, allowed to settle. The large quantities of sludge formed were filtered off and washed before discarding.

The removal of harmful impurities in the leach was carried out and the purified solution was received into another lead-lined wooden storage tank. This formed the feed solution, which was fed to the catholyte compartment of the cell, after passing through an intermediate feed tank. Sulphur dioxide gas was passed into

the solution until a concentration of 0.2 to 0.4 g./l. was obtained.

Electrolysis was carried out in a large, lead-lined, wooden vat provided with three stainless-steel cathodes and four lead-silver perforated anodes surrounded by cotton canvas diaphragms. The d.c. supply was from a 6-v., 350-amp. M.G. set. The temperature of the electrolyte was maintained in the range 30 to 33°C. by circulating cold water through lead coils immersed in the electrolyte. The spent anolyte from the anolyte overflow was collected in porcelain jars and sent back to the leaching tanks.

The stripping of the manganese metal is also an important step. If the surface of the cathode is too rough, it is difficult to strip the metal and a highly polished surface makes it difficult for the metal to adhere to the cathode. A semi-mirror polish is required to be given to the cathodes by buffing, followed by cleaning with nitric acid, washing with water and finally dipping for about 15 min. in a 2% solution of sodium silicate containing a small amount of caustic soda. Before stripping the metal, the cathodes were dipped in a solution of 1% potassium dichromate, washed and dried.

Appreciable quantities of electrolytic manganese metal have been produced with this unit and several pounds of this material have been supplied to research laboratories and other institutions. The purity of the metal produced is >99.9%.

A note discussing some considerations involved in the production of electrolytic manganese appeared in *CHEMICAL & PROCESS ENGINEERING*, 1954, **35** (3), 88.

**Gas and chemical plant.** An illustrated booklet describes in English, French, German and Spanish the activities of the Power-Gas Corporation Ltd. and its associate companies in the design and manufacture of plant and equipment for the gas, chemical, petrochemical, petroleum and iron and steel industries. Another is concerned with cyclic gas-making processes, and covers carburetted water-gas plant, blue-water-gas plant, complete gasification plant, high B.Th.U. oil gas plant and Segas catalytic oil-gas plant. A three-way valve, grit arresters, and automatic operation are also discussed. Two pamphlets from the same source deal, respectively, with the Segas catalytic oil-gas process and the Favorit filter process for the recovery of dusts and fumes from industrial processes.

# Studies on Catalysts Using Magneto-Chemical Techniques

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*The newer fundamental approach to the subject of catalysts has led to the use of experimental techniques the significance and potentialities of which may not readily be appreciated by many chemical engineers. In the article which appears below the author of our regular reviews of 'Catalysis' precludes his next review, which will appear in a forthcoming issue, by discussing magneto-chemical studies as an aid to the interpretation of catalyst structure and activity.*

**M**AGNETO-CHEMISTRY as such is not a new subject, but its potentiality as a tool in unravelling problems of structure have been developed only during the past 25 years. As emphasised in recent general works,<sup>1, 4, 6, 7</sup> its aim is to apply the results of measurements on magnetic properties to the solution of problems primarily concerned with structure. Applications in the field of catalysis are summarised in those more general works, and in three specific reviews by Selwood<sup>2, 3, 5</sup> covering work up to 1952.

Selwood<sup>2</sup> sees the principle problem as applied to catalysis as one of structural inorganic chemistry, 'to gather information about the identity, valence relationships, and atomic environment of the active substance.' It is not necessarily fortuitous that the elements and compounds which show catalytic activity—namely those of the transition groups in the periodic classification—are also those which show magnetic properties of a size and type amenable to study. This led to some conjecture, which Pink in 1949 felt to be still unresolved,<sup>7</sup> whether or not the intense local magnetic fields existing at the surface of catalytic solids could themselves have a catalytic effect—essentially by altering the quantum restrictions governing certain bond changes. However, Selwood emphasises that there is no experimental evidence for any general effect of the influence of an external magnetic field on chemical equilibria, that is, to an extent greater than 1%. There is some evidence<sup>2</sup> that the low-temperature catalysis of the ortho-para hydrogen conversion by strongly paramagnetic oxides such as chromia results from the intense non-uniform magnetic forces in the neighbourhood of paramagnetic surface ions acting on isolated hydrogen molecules.

The work of Hedvall's school in the 1930s on the reactivity of solids, comparing the catalytic activity of ferromagnetic materials as they passed from the ferromagnetic condition through the Curie temperature to the paramagnetic condition, had indicated two effects, known as the Hedvall effects, which received controversial support from later work. Recent careful work by Schwab and Goetzeller<sup>8</sup> confirms the first magneto-catalytic effect—that in the region of the Curie transition the catalytic activity is enhanced, due possibly to an unstable intermediate condition; the second effect, that in the paramagnetic condition the energy of activation for the particular reaction was lower than in the ferromagnetic condition, was not confirmed, suggesting that the magnetic condition itself has no direct effect on the active centres—which agrees with the absence of any influence of an external magnetic field.

We therefore conclude that there is no general magnetic effect in catalysis, and adopt the broad classification of magnetic methods in catalysis studies given by Selwood:<sup>5</sup>

(1) Use of paramagnetic susceptibility studies to obtain information about the active centres and their degree of dispersion. In this respect, changes in susceptibility accompanying adsorption processes can suggest mechanisms for electron transfer processes.

(2) 'Thermomagnetic' analysis, in which ferromagnetic components are investigated from points of view of their presence or absence, and of the influence of various factors on their Curie points.

## Fundamentals and origin of magnetic effects<sup>9, 1</sup>

Following Bates,<sup>9</sup> the field strength  $H$  is the magnetic intensity in oersteds in the absence of the magnetic material,

$I$  is the number of magnetic poles per unit area of the material (the intensity of magnetisation) and  $B$  is the magnetic induction, effectively the intensity per unit area within the substance in the presence or the field. Then we have:

$$B = H + 4\pi I$$

$$\text{or } B/H = 1 + 4\pi I/H$$

$$\text{or } \mu = 1 + 4\pi \chi$$

where  $\mu$  is the magnetic permeability and  $\chi$  the magnetic susceptibility per unit volume, or the intensity of magnetisation per unit field, alternatively expressible as  $\chi = \kappa/d$ , the magnetic susceptibility per unit mass (which, multiplied by the atomic or molecular weight, gives the atomic or molecular susceptibility). The specific magnetisation is  $\sigma = I/d$ , that is, the intensity of magnetisation per unit density. The magnetic moment  $M$  is the magnetic couple existing in uniform field of unit intensity, and may be measured in dyne cm./oersted or in Bohr magnetons, the Bohr magneton being the fundamental unit which is as real as the charge on an electron. The magnetic moment per unit volume is equal to  $4\pi I$ .

All substances exhibit some magnetic properties. The great majority are diamagnetic, that is, they show some resistance to the passage of the lines of force of the field through them, and so would tend to orient themselves diametrically to the field offering the least length to the passage of the lines of force; the permeability is thus less than unity, and the susceptibility is negative with a value of the order of  $10^{-6}$  e.m.u. A fair number, essentially elements of the transition groups or their compounds and 'odd electron' compounds, exhibit the reverse effect of paramagnetism, with permeability greater than unity, and susceptibility of the order of  $10^{-4}$  to  $10^{-5}$ . A further restricted number within this group

of substances exhibit *ferromagnetism*, which is a considerably stronger effect, strongly dependent on field strength and temperature, and giving way above a characteristic 'Curie' temperature to paramagnetic behaviour.

Diamagnetism is common to all materials, and therefore a correction for the inherent diamagnetism has to be applied in paramagnetic measurements. Its origin is in the production of induced magnetic dipoles by the action of the external field, the circulating currents from the orbital electrons producing magnetic moments in a direction to oppose the applied field. The susceptibility is independent of field strength and of temperature, and for a molecule is a reasonably additive property of the constituent atoms and certain structural values. It is of no direct interest in relation to catalysis.

Materials with permanent magnetic dipole moments will show paramagnetic effects due to orientation of these in the presence of a field, and it can be shown theoretically that the average magnetic moment will be directly proportional to the field strength and inversely to the absolute temperature. The moment arises from the orbital magnetic moment and from the spin magnetic moment of the electrons. However, in substances whose atoms or ions contain no partially filled electron orbitals, the orbital magnetic moments compensate to zero, as do the spin moments. Therefore, paramagnetism will be observed only with those substances containing partially filled atomic or molecular orbitals, such as the transition metals and their ions with partially filled *d*- or *f*-shells, or free radicals with a 'free' or odd valence electron.

The susceptibility of a paramagnetic material is primarily determined by distribution of electrons in the various allowed energy states and the Fermi distribution function, but for ions sufficiently separated to show little magnetic interaction the effective magnetic moment (which is primarily the spin moment) is approximately characterised by the number of unpaired electrons in the ion, being theoretically 0, 1.73, 2.83, 3.87, 4.90 and 5.92 Bohr magnetons for 0 to 5 unpaired electrons, respectively. This permits an estimate of the valence state of adsorbed ions. Further, any reaction which leads to filling or emptying of the incomplete shells by electron transfers should result in a change of overall susceptibility.

In contrast to these two effects, that of ferromagnetism is not an

atomic phenomenon. Rather it concerns groups of atoms constituting a 'domain' in which co-operative phenomena are displayed, leading to spontaneous magnetisation below the Curie temperature. This is due to electron exchange interactions between neighbouring atoms or ions which induce parallel orientation of the magnetic moments within the domain. The characteristic temperature variation of the specific magnetisation and the Curie temperature permit identification of ferromagnetic trace impurities in oxides or metals, and marked lowering or disappearance of the Curie temperature is indicative of high degrees of dispersion of ferromagnetic material. Further, reactions involving formation or removal of ferromagnetic material may be followed.

Certain oxides such as  $\text{Cr}_2\text{O}_3$ ,  $\text{NiO}$  and  $\text{CuO}$ , and many spinel systems, exhibit *antiferromagnetism*, characterised by a positive temperature coefficient of the paramagnetic susceptibility up to a characteristic temperature, above which normal paramagnetic behaviour is shown. This is attributable to exchange interactions between neighbours, resulting in an antiparallel orientation of the spins of neighbouring atoms or ions. Again, the phenomenon may be expected to disappear at relatively high degrees of dispersion, and so may give some insight into the state of supported material.

In the truly paramagnetic region, because of some exchange interaction effects still remaining, the temperature dependence of the susceptibility does not follow the Curie law ( $\chi$  proportional to  $1/T$ ) but the Curie-Weiss law ( $\chi$  proportional to  $1/(T - D)$ );  $D$  is the Weiss constant which is positive where there is a tendency to ferromagnetism and negative where there is a tendency to antiferromagnetism. Completely isolated atoms or ions, with negligible opportunity for any interaction with neighbours, might be expected to give  $D$  as zero, and the size of  $D$  may well reflect the degree of 'compactness' for a particular material, that is, the degree of dispersion.

#### Methods of measurement<sup>1-5</sup>

Except for the dynamic method involving measurement of periods of oscillation, which is little used, the methods depend basically on measurement of torsion forces or couples produced through the action on the sample of magnetic fields of up to 20,000 oersteds, usually obtained from electromagnets. A major difficulty, except

with ferromagnetics, is in the small values of the mechanical effects produced. However, adaptations of the Faraday type of magnetic balance have been designed, allowing accurate measurements on samples of around 25 mg. in apparatus permitting simultaneous adsorption or reaction rate studies.

As an example, one recent design<sup>10</sup> is suitable for thermomagnetic work and allows an automatic record to be made of the force required to keep the sample in its original position, that is, to counterbalance the magnetic forces developed, as the sample temperature is altered. The field from the electromagnet acts through a furnace tube on the sample in the horizontal quartz tube passing through a brass block suspended from a torsion wire. Under the influence of the varying forces (for example, as the temperature is raised) the tube swings between the make-and-break contacts (adjusted to allow only a fraction of a mm. motion), which activate a reversible motor, applying through a worm and torsion head a torque to the torsion wire, which tends to restore the sample to its original position, when the contact will be broken. A motor shaft connects through the worm to a *Helipot*, the e.m.f. produced from which, through the turning of the motor, being applied to a *Speedomax* recorder to give a constant recording which will be proportional to the intensity of magnetisation. The magnetic pole pieces are specially shaped to produce the inhomogeneous field in the direction of motion required in the Faraday method.

In another apparatus, the sample hangs from a silica spiral balance in a vertically graded field (special pole pieces), which is accurately mapped using a specimen of known susceptibility and altering its position by moving the whole assembly relative to the magnet with the aid of the mechanical stage. The force acting on the sample is proportional to its mass, its susceptibility, the field strength and the field gradient. This force produces an apparent increase or decrease in weight of the sample, that is, an extension or contraction of the spring which can be accurately measured with the micrometer microscope. A furnace tube can be inserted around the sample to vary the temperature, and suitable modifications may be made to the glass assembly to permit ancillary studies such as effects of altering the atmosphere surrounding the sample (adsorption), or of reduction of the sample.



## Effect of adsorbed layers on magnetic properties

Perhaps because of the experimental difficulties of differentiating between bulk and surface effects, there is relatively little direct information on the effect of adsorbates on the catalyst adsorbent. The literature to 1949 was reviewed by Pink.<sup>7</sup> Omitting cases where the active catalyst is supported (that is, dispersed whether by adsorption or not) on inactive material, the only direct evidence then concerned the effect of catalyst poisons for hydrogenation over Pd catalysts. Using very fine powder, Dilke, Eley and Maxted had shown that adsorption of dimethyl sulphide lowered the susceptibility to an extent indicative of the transfer of one electron from each adsorbed molecule to the palladium. This, coupled with the observation that only those metal ions possessing *d*-orbital electrons could poison hydrogenation catalysts such as Pt and with work on the relation between catalytic activity and paramagnetic susceptibility of alloy systems, led to the development of current theories on metal hydrogenation catalysts recently reviewed.<sup>12</sup>

It is known that, with those metals capable of taking up large quantities of hydrogen in solution (Pd, Ti, Zr, etc.), there is measurable decrease in the paramagnetic susceptibility. Similar effects with adsorbed layers of hydrogen on metals with incompletely filled *d*-bands of electrons should be observable if the ratio of surface to bulk atoms is high enough.

Selwood, Adler and Phillips have recently shown<sup>13</sup> that with highly dispersed nickel on silica at nickel particle diameters of the order of 50 Å or less, exposure to hydrogen decreases the specific magnetisation of the nickel (compared with the value in helium or vacuum) to an extent which increases with increasing degree of dispersion. There was a similar effect with ethylene, indicating electron donation in both cases to the incompletely filled 3*d*-band of nickel associated with its ferromagnetism. The opposite effect was observed with oxygen, and possibly nitrogen, indicating acceptance of electrons from the nickel by these atoms. They consider this approach a powerful tool for studying electronic transfers accompanying chemisorption on metals.

Reyerson and Honig<sup>14</sup> investigated the diamagnetic susceptibility of nitrogen dioxide adsorbed on rutile, making a constant correction for the paramagnetism of the substrate. There was an apparent fall from  $-1.5$  to

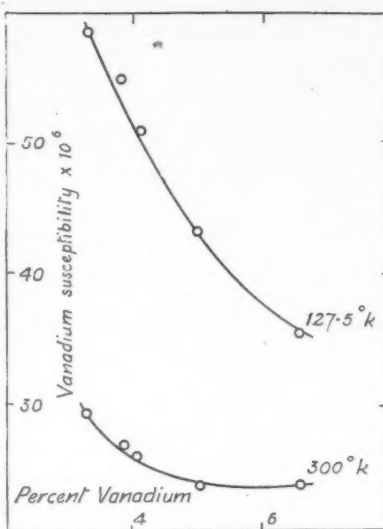


Fig. 1. Magnetic susceptibility isotherm for vanadia supported on rutile (Selwood and Lyon<sup>13</sup>).

$-0.5 \times 10^{-6}$  as the degree of surface covering increased. On the basis of Ehrlich's previous data showing an increase of paramagnetic susceptibility for titanium dioxide with increasing oxygen deficiency, they take this as evidence that their rutile sample had oxygen defects and that these were progressively re-oxidised on adsorption of the gas. This work suggests that investigations of the effect of adsorbed gases on magnetic properties of the surfaces of defect oxides, along the line of those on surface conductivity,<sup>19</sup> would be helpful.

## Structure of supported oxides

Selwood and co-workers<sup>1-5, 18</sup> have investigated a number of transition oxides supported on high-area carriers such as alumina, titania and silica, by means of susceptibility isotherms of the form shown in Fig. 1, where the susceptibility per gramme of supported material is plotted as a function of its concentration. These isotherms are in general characterised by a strong increase with decreasing concentration—indicative of magnetic dilution or dispersion—but their interpretation varies in individual cases. The broad picture which emerges is that the active oxides are distributed at areas over the surface with bare patches of support in between. The support may in certain cases (iron, manganese and nickel on alumina) 'induce' its cation valency on the supported cation, resulting in a supported oxide of mixed cation valency—which appears to be associated with eventual high catalytic

activity of the system after reduction. There is evidence of ferromagnetism in certain oxide phases.

A recent re-investigation of copper oxide on gamma alumina<sup>11</sup> at copper concentrations down to less than 0.5% confirmed the general trend, and showed that even at the lowest concentrations the reduced copper was diamagnetic and so could not exist as isolated copper atoms—which would be paramagnetic. Further, on re-oxidation after prolonged 'sintering' at temperatures known to lead to loss of catalytic activity, the original susceptibility isotherms are obtained. This shows that there cannot have been a true sintering in the sense that the copper atoms migrated thermally to build larger agglomerates, as these would on re-oxidation have given larger agglomerates of oxide with lower susceptibility per gramme.

Kobozov and co-workers<sup>17</sup> reported studies on the paramagnetic susceptibility as a function of dilution on the support for a number of metals normally paramagnetic, ferromagnetic or diamagnetic in the bulk phase, by a method claimed to have excluded ferromagnetic effects. The ratio *f* of the susceptibility per gramme adsorbate to the susceptibility of the bulk material was found to increase markedly with decrease of the degree of surface covering *a* (estimated from the surface area of support and area occupied by adsorbed atom) for Co and Ni salts on silica gel and carbon. For Pt and Fe on alumina gel the *f* values were extremely high at *a* less than 0.005, but the susceptibility was substantially lower than for ferromagnetism and independent of field strength. They suggest the occurrence of 'super-paramagnetism' in these adsorbed stages, due to marked enhancement of the orientation of adatoms. Silver on barium carbonate is considered to show the same effect. For the hydrogenation of ethylene, there was a definite correlation of catalytic activity with susceptibility per gramme active Pt on various types of carriers—the activity tended to a maximum as *a* approached zero, as did the susceptibility (electron accepting catalyst). On the other hand, the catalytic activity of supported silver for hydrogen peroxide decomposition decreased with increasing paramagnetic susceptibility (indicating an electron donating catalyst).

This work is based on the concept of mono- or diatomic dispersion at great dilutions, which is contrary to Selwood's concept of patches of active material, as discussed below.

## Thermomagnetic analysis and ferromagnetism

As already indicated, measurements of Curie points offer a means of controlling or following changes in content of ferromagnetic substances.<sup>1-5</sup> The facts that the Curie temperature appears to be dependent on the coordination number of nearest neighbours and to vary with ratio of surface to bulk atoms within a particle, have suggested the possibility of estimating particle size distributions for ferromagnetic metals in disperse states.<sup>13, 16</sup>

Heukelom, Broeder and Reijen,<sup>16</sup> from a magnetic study of nickel on silica supports as a function of reduction time, have suggested that the reduction process consists first of reduction, then of sintering and recrystallisation, the former with approximately half the activation energy of the latter. The magnetic measurements, supported by adsorption data, indicate reduced nickel particles of average diameter around 50 Å, that is, containing about 10,000 nickel atoms.

The results of Selwood *et al.*<sup>13</sup> are substantially similar. They emphasise the need for caution in estimating the extent of reduction of supported catalysts simply by comparing the expected and observed ferromagnetism developed per gramme total metal in the catalyst. The effect of sintering is strongly brought out in thermomagnetic curves, from which it is obvious that extensive reduction may occur without development of ferromagnetism, whereas subsequent sintering eventually develops the characteristics of massive nickel. The nickel content of the catalyst was about 42%, and it is interesting to note that a nickel film of about 0.3 g./sq.cm., in contrast, gave the magnetic curve for massive nickel. Once a sample was sintered, re-oxidation and reduction gave the original curve, indicating that the differences were in the initial dispersion of the first-reduced nickel.

From the form of the magnetisation curves, estimates of particle size as a function of sintering temperature were obtained, and were in substantial agreement with those from x-ray line width broadening. At 450°C. sintering temperature, there was a marked drop in catalytic activity for benzene hydrogenation, incommensurate with the observed fall in specific surface (adsorption measurements), which suggests the existence of an intense activity factor over and above the surface effect.

As a final example of another approach, Westrick<sup>15</sup> has reported briefly on studies on the Curie points of mixed iron catalysts of interest for

ammonia synthesis. Addition of alumina to magnetite followed by melting gave products with Curie points decreasing from 570° for pure magnetite to 500°C. at an Al content equal to 30% of the total trivalent ion, after which there was no further decrease. This supports the indications from work on spinel structures that the ferric ion in magnetite is fairly easily replaced by aluminium, and also earlier results obtained by co-precipitation of Al with ferric ions followed by reduction of the mixed oxides at 400°C.

## Conclusion

The above examples serve within the limitations of a short review to illustrate the potentialities of, and current trends in, the application of magneto-chemical methods to the study of problems relating to catalysts. It is by no means yet clear what technical advances may result from such studies. However, just as Hauffe<sup>19</sup> foresees mutual benefit from investigations in the catalytic and electro-technical industries from the standpoint of electronic conducting properties of materials of common interest, so we may expect results of common

interest to emerge from magneto-structural studies on catalysts and on oxide systems of the spinel and other types in relation to their use as high-frequency core materials.

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## Polythene's Progress

WORLD output of polythene today is about 150,000 tons p.a. and it is predicted that by 1965 the world demand will have increased to more than 1½ million tons p.a. This is stated by A. Renfrew, B.Sc., in a paper presented to the Manchester Association of Engineers. Recalling that polythene owes its discovery to pure research work on chemical reactions, he points out that this research work took place at a time when industry was suffering from one of its periodic slumps. In the chemical industry, as in the engineering industry, a period of recession is often an excuse for cutting down research and development work. There is a good deal of evidence to show that exactly the opposite policy would be the right one. Certainly, he says, it seems to pay off well now and again.

The properties of polythene in the molten form are of vital importance for the fabricator who has to shape the hot melt by moulding or extrusion, and a knowledge of the molecular weight, molecular weight distribution and chain branching are necessary if the melt properties are to be properly controlled. Since many of the solid

properties are also related to the structure of polythene, an accurate knowledge of melt viscosity is a great help in selecting specific polythenes for specific uses. A simple piece of apparatus called a grader was developed in the United Kingdom to determine melt viscosity or melt flow index and has now been adopted internationally. It is an extrusion plastometer in which the weight in grammes extruded in 10 min., under controlled conditions of temperature and pressure, is measured.

The chemical resistance of polythene is outstandingly good. Indeed, it has been described as complementary to that of metals, since most of the reagents which attack metals have little effect on polythene at room temperature. If articles are improperly fabricated, chemical resistance may appear poor. A sure indication is the elongation at break; if this is lower than normal for the type of polythene being used, the conditions of fabricating have been wrong.

Polythene pipes have found important uses in chemical plants; polythene is being specified for many chemical plant applications where

resistance to cold or cool acids and alkalis is required. Trade skill in manipulating it has been highly developed and in the United Kingdom it is probably the most important plastic used in chemical plant.

It has often been asserted that rats attack polythene. This is the reverse of the truth. Wild rats were kept for some time without food in a cage with bars from common pipe materials, but, in their attempts to escape, left polythene alone.

The processing of polythene usually consists in shaping the hot melt and holding it in the required position until cool. The extrusion process is used for film and pipe and most of the heat is supplied in modern machines by the mechanical work when the screw is rotated inside the barrel.

Rigid polythene pipe is manufactured using an extruder similar to that used for film, but the haul-off and cooling arrangements are quite different. The problem of this type of extrusion is to maintain accurate dimensions, and a whole variety of sizing dies have been developed for this purpose. Recently, extruders with two or more screws have been marketed. The heat is supplied more by conduction from external heaters than by mechanical work and the barrels are consequently short. Tubes are also made by centrifugal casting, a process analogous to that used in the metal industry. The maximum length so far made is about 12 ft. and the maximum internal diameter about 3 ft.

### Taking the air out of paper stock

A de-aeration process called the *Deculator* has been developed to solve the problem of trapped or dissolved air which gets into paper stock during the papermaking process. It is claimed that all air is completely removed from the stock just before it enters the headbox of the fourdrinier.

In the *Deculator*, paper stock flows through a distribution header into a large cylindrical receiver. The interior of the receiver is held at a high vacuum close to the vapour pressure corresponding to the boiling point of the stock. Large nozzles whirl the stock at high velocity and spray it against impingement areas. Liberated air is then removed by vacuum pumps and the de-aerated stock is pumped to the headbox of the paper machine. The process is continuous and automatic.

The process has been described in a technical bulletin of the International

Nickel Co. Inc., which reports that selecting proper materials of fabrication for the *Deculator* receiver assembly presented a two-fold problem. First, there was the corrosive action of the paper stock. Not only did the metal used have to stand up under that attack, but it had to resist the constant erosive impingement of the spraying action within the de-aerator. The Rotaread Corporation, Bronxville,

N.Y., who manufacture the *Deculator*, experimented with many materials, but found chromium-nickel stainless steel types 316 or 304 the best answer.

The de-aerator operates successfully on many grades of paper, including newsprint, book, kraft, multi-wall bag, gumming, and butter carton. Among other advantages, it allows a reduction in sulphite content in newsprint stock without reducing machine speed.

## Separating Mixtures of Liquids —a New Apparatus

A NEW development of solvent-extraction technique is embodied in a rotary-disc apparatus which was constructed for the N.V. de Bataafsche Petroleum Maatschappij by the Koninklijke Shell-Laboratorium, Amsterdam. This apparatus will be of interest to those branches of the chemical industry where solvent extraction is employed.

The aim was to build an apparatus working on the counter-current principle, which had a great number of mixing compartments, gave a good extraction yield, was of simple construction and was of a size which would not make the whole process uneconomical. The apparatus consists of a vertical cylinder, which is divided into compartments by means of horizontal rings fixed to the wall of the column. In the middle of each compartment a horizontal disc rotates at a speed which can be varied. The diameter of the discs, which are fixed to the vertical shaft of the column, is somewhat less than the diameter of the rings separating the compartments. The discharge of the denser liquid is regulated by a level controller; the discharge of the less dense liquid by a pressure controller. There have been no difficulties in building units for dealing with corrosive liquids and for pressures up to 40 atm.

The less dense liquid enters the counter-current extraction column at the bottom, the denser liquid at the top. One of the two liquids is the dispersion medium, the other the disperse phase. If the denser liquid is the continuous phase, the less dense liquid ascends in form of small drops. The boundary between the phases, i.e. the place where the drops unite, is in the upper part of the apparatus just above the inlet for the denser liquid. If, however, the less dense liquid is the continuous phase, the denser liquid descends in form of drops and the boundary between the phases

is in the bottom part of the column just below the inlet for the less dense liquid. The boundary is therefore never between the two inlets, and the liquids disperse and reassemble only once. This saves much time.

Centrifugal forces play an important part in the formation of currents caused by the rotation of the discs, but these currents are, of course, influenced by the fact that the two liquids pass through the column in opposite directions. The size of the drops can be regulated by altering the speed of rotation.

The apparatus has been described in detail by G. H. Reman and J. G. van de Vusse in *Chimie & Industrie (Suppl. Mensuel)* for October 1955. Many experiments have been made to find out how efficiency and yield vary with the speed of rotation, the diameter of the discs, the opening of the rings, the height of the compartments, the diameter of the column and the quantity of one phase in proportion to the other, and the apparatus has been used with success for laboratory extractions as well as for extractions on an industrial scale, such as the extraction of acetic acid from methyl-isobutyl ketone with water, the extraction of *n*-butyl amine from kerosine with water, the purification of synthetic detergents, the extraction of lubricating oil by means of furfural and the extraction of mercaptans from petroleum fractions.

A separation which has been studied in great detail is the extraction of lubricating oil by means of furfural. The purpose of this extraction is the removal of aromatic components from the lubricating oil in order to increase its viscosity. The furfural enters the apparatus at the top and the lubricating oil at the bottom. For this separation as well as for many others, it is stated, the rotary-disc contactor has proved to be more economical than any other device.



# British Patent Claims

The following are abstracts of some recent British patents which will be of interest to our readers. These abstracts are reproduced from the weekly Patents Abstracts Journal by permission of the Technical Information Co. The complete specifications can be obtained from the Patent Office, 25 Southampton Buildings, London, W.C.2, price 3s. each.

## Sealing gasket

A sealing arrangement for high-pressure fluids (e.g. in a screw-down valve assembly), in which a closure member for an aperture has a shoulder of conical form confronting an abutment surface, comprises an annular gasket under pressure in the aperture between the shoulder and surface, such gasket having a central metal core with a coating of softer metal thereon. The inner surface of the gasket is of conical form to engage the shoulder.—739,491, *Edward Valves Inc. (U.S.)*.

## Grinding mill

A ball or like grinding mill, having an inlet at one end and an outlet at the other end, has a lining (e.g. helically grooved) which over part only of the axial length of the grinding chamber tends to classify the grinding bodies by bringing about movement of the smaller and larger grinding bodies in opposite directions to one another. The invention may thus be applied to prevent accumulation of the smallest grinding bodies at the outlet.—739,578, *F. L. Smidth & Co. A/S. (Denmark)*.

## Removing slag from a metallurgical converter

A device for loosening and/or removing slag comprises a manually controllable piston, within a cylinder, for striking a rod carrying a slag contacting tool. When operating, an initial working stroke of the piston pushes the rod until the tool bears against the slag, and after each return stroke the piston subsequently strikes the rod and the impact is transmitted to the tool for loosening the slag.—739,674, *Pintsch Bamag Akt.-Ges. (Germany)*.

## Nitric acid

Production of  $\text{HNO}_3$  having a concentration of the order of 99.5% and freed from  $\text{N}_2\text{O}_4$ . Impure acid, having a concentration  $> 68\%$ , is fractionated in a column the base of which is at 120 to 125°C., and vapours containing  $\text{HNO}_3$  and  $\text{N}_2\text{O}_4$  taken off from the head of the column are introduced

into a second column, the base of which is at 86 to 90°C. Pure anhydrous  $\text{HNO}_3$  is withdrawn from the base of the second column.—739,482, *M. E. J. Cathala (France)*.

## Liquid-suspension separator

A dense-liquid centrifugal device, for separating particles (e.g. coal, ore etc.) in suspension, incorporates a unit consisting of two wholly cylindrical concentric tubes to which the material is supplied by a spiral conduit located in the upper part of the space between the tubes.—739,705, *Preparation Industrielle des Combustibles (France)*.

## Solvent extraction apparatus

An apparatus for solvent extracting oils, etc., from solids in which solids-retaining cells are disposed angularly around an upright axis (about which the cells advance in a closed horizontal path) and pass beneath solvent sprayers, at least some of which are of elongated form and are disposed above the cell path and transversely thereto, these sprayers being adjustable so that the place at which solvent enters a cell can be varied along the path. At a given point in the path the cells are charged with solids, and at a second point in the path the extracted solids are discharged from the cells.—739,602, *Blaw-Knox Co. (U.S.)*.

## Processing of perlite, etc.

Process for heat-treating perlite and similar minerals to produce very lightweight aggregates of considerable crushing strength. The crushed material is introduced into the top of a vertical furnace into the flame zone of the furnace burner; it is then conveyed down a vertical, open-ended primary heating chamber within the furnace, circulated from the lower part of the furnace up through a secondary chamber formed between the primary chamber and the interior furnace wall and communicating with the top of the primary chamber, and the material is deflected back into the flame zone in the primary chamber and recirculated till the desired degree of expansion is achieved. The product is finally discharged at the top of the furnace. A furnace for such a process is claimed.—739,617, *J. J. Clift (Australia)*.

## Carbonisation plant

A retort for carbonising coal, etc., by direct internal heating has a grate provided with a central, upwardly

tapering projection, and means for raising the grate into a working position in which the projection lines within the carbonisation space, and for lowering it into a position in which the retort contents can be discharged by gravity. The conical projection occupies the space which would otherwise be occupied by incompletely carbonised core.—739,717, *National Carbonising Co. Ltd.*

## Electrolysis apparatus

In a system for the decomposition of salts of organic acyclic acids (to produce and recover useful products) an anode contacts a liquid electrolyte containing the acid resulting from electrolysis of such salt, a cathode contacts an electrolyte containing the resulting cation, and these electrodes are separated by a permselective membrane wet with electrolyte. The electrode compartments are provided with means for introducing an electrolyte, and an outlet for recovering a gaseous product separately.—739,722, *Hooker Electrochemical Co. (U.S.)*.

## Rubber production

Production of high-grade rubber from the skim portion left after separating a rubber-rich cream portion from a natural rubber latex. The skim portion is allowed to spontaneously coagulate and the coagulum is finely divided, treated with an aqueous alkali metal or ammonium hydroxide solution, and then washed and dried.—739,750, *Firestone Tire & Rubber Co. (U.S.)*.

## High-pressure pump

In a reciprocating pump for generating pressures above 1,000 atm. (particularly for use in chemical processes, e.g. ethylene polymerisation), a single pump-chamber cylinder reciprocates relative to a stationary plunger-type piston carrying suction and delivery leads with valves at their inner ends. The chamber has walls of uniform thickness closed by a hemispherical end also of uniform thickness. In B.P. 736,664 a reciprocating cylinder has two chambers of two different diameters, with a stationary piston in each chamber. Inlet and outlet ducts are in the larger and smaller pistons, respectively, and a duct carrying a non-return valve is provided in a wall separating the two chambers.—736,583 & 736,664, *Imperial Chemical Industries Ltd.*

## ★ Personal Paragraphs ★

★ **Lord Strathalmond**, C.B.E., has indicated that he wishes to retire from the chairmanship of the British Petroleum Co. Ltd. on March 31, when he will have completed 33 years' service as a director of the company—28 years of this as chairman or deputy chairman. **Mr. B. Jackson**, the present deputy chairman, is to be chairman, and **Mr. N. A. Gass**, C.B.E., M.C., is to be deputy chairman, from April 1. The **Hon. M. R. Bridgeman**, C.B.E., will be appointed to fill the vacancy on the board caused by Lord Strathalmond's retirement.

★ After 30 years as joint managing directors of John & E. Sturge Ltd., **Mr. A. L. Wilson** and **Mr. A. P. Wilson** have relinquished their positions. They are continuing their interests on the board of directors as chairman and deputy chairman, respectively. **Mr. A. R. Foxall** and **Dr. E. R. S. Winter** have been appointed joint managing directors.

★ At the annual general meeting of the British Tar Confederation the following were elected as officers of the confederation for the year 1955-56: **Sir Harold C. Smith**, K.B.E., D.L. (president); **Mr. L. W. Blundell** (honorary treasurer); **Mr. Henry F. H. Jones**, M.B.E. (chairman of the executive board); **Mr. R. H. E. Thomas**, O.B.E., and **Mr. Stanley Robinson** (vice-chairmen of the executive board).

★ The appointment of **Dr. T. P. Hughes** as director of the Tube Investments research laboratories at Hinxton Hall, Cambridge, has been announced. **Dr. F. P. Bowden** is the chief research consultant to Tube Investments Ltd.

★ **Sir Edward C. Bullard** has resigned from the directorship of the National Physical Laboratory.

★ **Mr. F. E. Mills**, until recently sales director and general manager, has been made managing director of B.I.P. Engineering Ltd., Streetly, Staffs.

★ **Mr. D. P. C. Neave**, vice-chairman of the Consolidated Zinc Corporation Ltd., has been elected chairman of the Zinc Development Association. The former chairman, **Mr. William Mure**, managing director of the British Metal Corporation Ltd., will continue as a member of the Council of the Association.

★ We regret to announce the sudden death, on January 2, of **Mr. S. J.**



Mr. G. A. Dickinson.



Mr. J. N. M. Legate.

**Matthews**, head of the research and development division of Baird & Tatlock (London) Ltd. **Sir Bernard Keen** is temporarily taking charge of the division.

★ **Messrs. K. A. Hogan, W. H. Sharland and D. G. Badham** have been appointed directors of Powell Duffryn Ltd.

★ **Mr. Miles B. Reid** has resigned his directorship of Stephenson Clarke Ltd. to enable him to devote more time to his duties in connection with his directorships of Powell Duffryn Ltd., Mobil Oil Co. Ltd. and other companies.

★ **Mr. Hierman Hirst**, buying manager of the chemical section of the central purchasing department of Imperial Chemical Industries Ltd., has retired after 40 years' service.

★ **Mr. J. G. Window**, sales director of Q.V.F. Ltd., recently returned from a seven-day visit to Finland with £5,000-worth of orders. During his 1,000-mile tour he visited several companies using his firm's products.

★ **Mr. R. Hart Still** has been appointed to the board of Bakelite Ltd. The company has announced a number of additional new appointments as a result of the reorganisation of the sales and production departments.

★ **Mr. D. A. Strachan** has been appointed to the head office of the General Electric Co. Ltd. and will be responsible for flameproof and mining equipment sales. **Mr. S. C. Wells** has simultaneously been appointed to take responsibility for oil industry equipment sales.

★ **Mr. K. J. B. Wolfe** has been appointed head of fundamental research by the Triplex Safety Glass Co. Ltd. and will take charge of the company's new research department to be built at their Willesden factory in West London.

★ **Mr. H. E. Charlton** has resigned from the board of Petrocarbon Developments Ltd. in order to devote all his time and energy to H. E. Charlton Engineers Ltd.

★ On account of the increasing pressure of his duties in the Powell Duffryn group and elsewhere, **Sir Henry Wilson Smith**, K.C.B., K.B.E., has resigned from the board of Mobil Oil Ltd.

★ **Mr. R. B. Killingsworth**, manager of Mobil Oil Co.'s technical department since 1952, returned to the United States recently. He has been succeeded by **Mr. G. A. Dickinson**.

★ **Mr. J. N. M. Legate** has been appointed assistant chief engineer of the industrial control department of Metropolitan-Vickers Electrical Co. Ltd.

★ **Mr. F. B. Howard-White**, M.C., has been appointed a member of the delegate board of the Mond Nickel Co. Ltd. He was appointed director of Henry Wiggin & Co. Ltd. in May last year; in recent years has travelled abroad extensively on important missions for the parent company.

# World News

## GREAT BRITAIN

### Works training of chemical engineering apprentices

Guidance on the practical training of the increasing number of young men who are entering the profession of chemical engineering through apprenticeship schemes is provided in a note that has been issued by the Institution of Chemical Engineers. This note points out that the object of a student apprenticeship in chemical engineering should be to produce an all-round chemical engineer rather than a specialist in any one branch of the industry. It outlines a scheme of training, giving tentative suggestions as to the periods which might be devoted to each subject.

### Furnace refractories discussion

Under the auspices of the Institute of Metals, an all-day informal discussion on 'Furnace Refractories for the Use of the Non-Ferrous Metal Industries' will be held at the University, Edgbaston, Birmingham 15, on Friday, February 24, at 10.30 a.m.

### Careers in atomic energy

The United Kingdom Atomic Energy Authority has issued an attractive little booklet explaining its apprenticeship scheme. Facilities are available for training in engineering (electrical and electronic, or mechanical or chemical, or physics) and in metallurgy. The booklet contains a foreword by Sir Edwin Plowden, K.C.B., K.B.E., chairman of the Authority, and numerous photographs illustrating the work done by apprentices.

### Price reduction

As selling agents for British Petroleum Chemicals Ltd., British Industrial Solvents have announced a reduction of £25/ton in the price of dicyclopentadiene.

### Atomic energy agreement with India

Discussions between the United Kingdom Atomic Energy Authority and the Indian Department of Atomic Energy have led to the conclusion of an agreement which ensures that there shall be close co-operation and mutual assistance between the Authority and the Department in the promotion and development of the peaceful uses of atomic energy. The agreement provides for the Authority and the Department to arrange for members of their staffs to consult and work

together on mutually agreed topics.

In furtherance of this agreement the United Kingdom Atomic Energy Authority will provide the Indian Department of Atomic Energy with enriched uranium fuel elements for a swimming pool reactor now under construction at Bombay. The agreement also includes arrangements for the Authority to assist in the design and construction of a high-flux research reactor which may be built at a later date.

### Instrument centre

A permanent exhibition by member firms is being opened by the Scientific Instrument Manufacturers' Association at their London headquarters.

### Fighting fires in oil tanks

A method of fighting fires in oil tanks has been developed, utilising air or other gas injection to induce upward flow from the colder sub-surface body of the oil layer and thereby to cool the surface of the burning liquid. This cooling is sufficient to extinguish fires of high-flash-point oils and to reduce the severity of fires of low-flash-point oils so that they can be readily extinguished by conventional means.

The effectiveness of the air agitation method has been demonstrated on fires of petroleum and a variety of petroleum products in both small and large tanks. Extinguishment or control is usually obtained within a few minutes. The method is applicable to contained fires of any petroleum liquid having a Reid vapour pressure of 13 lb. or less.

Complete extinguishment can be obtained of contained fires of kerosene, diesel oil, fuel oils, transformer oils, lubricating oils and distillates; and, under special circumstances, asphalt and high-flash-point crude oils. The intensity of fires of gasoline, naphtha and low-flash-point crude oils can be reduced to the point where they can be readily extinguished by other means.

A film dealing with this method of fire fighting was shown in London recently by the Mobil Oil Co. Ltd.

### Laboratory glassware for Turkey

Chemistry students at Ankara University (Turkey) will soon be using British laboratory apparatus supplied by Quickfit & Quartz Ltd. This will be the firm's first major export to Turkey.

## British Productivity Council officers elected

General appreciation of the work done by Sir Ewart Smith during his year as chairman was expressed at a recent meeting of the British Productivity Council at which officers were elected for the coming year. Mr. James Crawford, general president of the National Union of Boot and Shoe Operatives and a member of the Trades Union Congress General Council, was elected chairman. Sir Ewart Smith will retain his seat on the Council.

The Rt. Hon. Lord McCordale of Newton, P.C., who was recently appointed a vice-president of the British Employers' Confederation, was elected deputy chairman of the B.P.C. Dr. W. H. Garrett, M.B.E., J.P., a director of Monsanto Chemicals Ltd. who is also a vice-president of the B.E.C., was elected to the Council. Sir Colin Anderson and Mr. W. T. Winterbottom, C.B.E., resigned.

### Cement-making plant for Iran

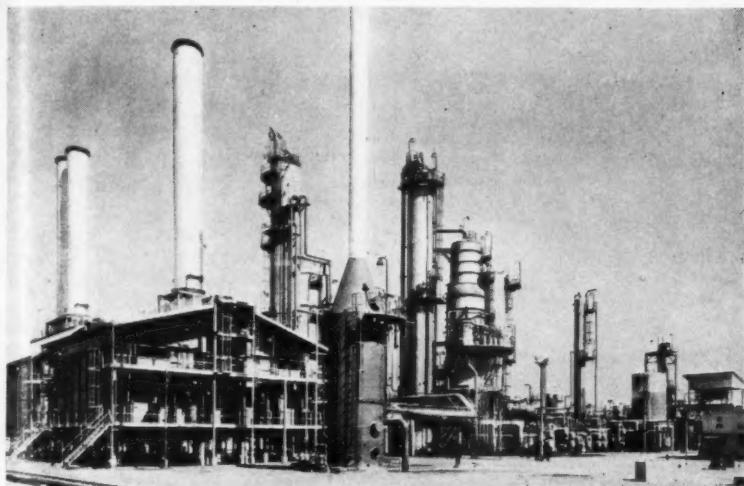
Edgar Allen & Co. Ltd. have announced that they have signed a £3-million contract with the Seven-Year-Plan Organisation of Iran for the supply of cement-making machinery and equipment. This is for the proposed Portland cement factory at Doroud, 250 miles south-west of Teheran. The plant will produce 600 tons/day of Portland cement. It is understood that this is the first instance since the war of an order for a plant of this description being secured in the face of international competition.

The equipment, which includes two rotary kilns 330 ft. long by 9 ft. and 10 ft. 6 in. diameter, primary crusher, wash mills, tube mills for raw and finished materials, together with all ancillary equipment, will be fabricated at the Imperial Steel Works. The plant consists of two units, delivery of the first to be completed in 18 months, and the second six months later.

### Ventilation in industry

Where there is a major ventilation problem in a building, this must be treated before dealing with general ventilation. For instance, you may have large quantities of steam, smoke, heat, etc., and these must be treated at the source; once the trouble is allowed to spread, it becomes a major problem. Imagine a private sitting-room with a fireplace; ventilating this room is an easy task, but if the chimney were blocked up and all the smoke were allowed out into the room, then it would be a major problem getting





Furnaces for combination topping and cracking unit at Iraq refinery.

rid of the smoke as well as providing proper ventilation.

This example comes from a talk given at the Building Exhibition in London recently by Mr. M. J. Reaney, of Colt Ventilation Ltd. He pointed out that, having decided on the steps to be taken to obtain the conditions desired and the number of air changes necessary, the next question was what type of equipment to use—should it be extract fans in the roof and natural inlet at low level, or inlet fans at low level with high-level natural extract? Whenever fans were necessary, the second method was to be recommended, because a fan always sucks its replacement inlet air from the nearest opening, which so often is from under the eaves or the joints in roof sheets. While this allows perfect ventilation in the roof space, it may leave the air completely stagnant at working level, but when air is blown in with fans and extracted naturally from the building under a plenum, greater flexibility resulted.

But why use fans at all when you have got a problem of excess heat, asks Mr. Reaney? Thermal currents of considerable horsepower and stack velocity in hot industry, all going to waste and causing discomfort to operatives, could be harnessed and increased by up to 25% by the use of properly designed natural roof extractor ventilators to give a power of extraction equal to fans, and all without maintenance or running costs.

#### FRANCE

##### Polyethylene plant

Two of France's leading chemical concerns, Societe des Usines Chim-

ques Rhone-Poulenc, and Etablissements Kuhlmann, have decided to build a plant to produce polyethylene under a process developed by the Phillips Petroleum Co., according to a news agency report.

#### EIRE

##### Fertiliser projects

Plans to establish two new fertiliser factories in Eire—one in Cork and the other in Sligo—have been announced by W. & H. M. Goulding Ltd., of Cork.

The Cork plant is scheduled to produce 50,000 tons p.a. of compound fertiliser and the same quantity of superphosphate. It is planned to erect a second plant in Cork later to produce an additional 50,000 tons of superphosphate. In Sligo, 20,000 tons p.a. of superphosphate will be produced.

The company, which is already manufacturing agricultural fertilisers, will seek a State grant for the Sligo project.

Rumours have been current that Dutch and German firms have been invited to set up fertiliser manufacturing plants in Eire.

#### FINLAND

##### Vanadium from magnetite concentrate

A new method for separating vanadium from magnetite concentrate is being used by the Otanmäki Co., Finland's youngest mining company. As a first step the magnetite concentrate is pelletised by adding Glauber salt. In the course of sintering the vanadium oxide reacts with the salt, forming a soluble compound which is then dissolved in water. The solution

is finally treated with sulphuric acid; the precipitate is smelted and sold as flakes containing 90 to 94%  $V_2O_5$ .

The new process has been discussed by M. Merenmies in *Teknillisen Kemian Aikakauslehti* recently. The vanadium plant now being erected will commence operations next summer with an annual capacity of 70,000 tons of pellets and 600 tons of vanadium pentoxide. Later the capacity will be doubled.

#### YUGOSLAVIA

##### Cellulose works

Production tests have started at the Yugoslav sulphate cellulose and kraft paper works in Maglaj, which is expected to be in full production later this year, with gross annual output valued at 5,000 million dinars. Reports state that the factory, which is entirely equipped with Yugoslav machinery, will obviate the necessity for further imports of cellulose and kraft paper.

#### IRAQ

The first complete oil refinery in Iraq, built for the Government near Baghdad, is now on stream. Designed and engineered by the M. W. Kellogg Co., the new 24,000-bbl./day thermal refinery processes Kirkuk crude to produce fuel gas, stabilised gasoline, kerosene, light gas oil, diesel oil and residual fuel oil.

A single combination unit is responsible for this production—integrating crude atmospheric topping, single-pass visbreaking of reduced crude, thermal cracking of gas oil, thermal reforming of heavy virgin naphtha, vacuum flashing of combined evaporator bottoms, gas recovery, and the stabilisation of combined gasoline. Complementing the combination unit are gasoline treating and kerosene treating units, a complete power plant and auxiliaries.

Two unique features are the plant's sulphuric acid and can-manufacturing facilities. The sulphuric acid plant will reclaim spent acid as well as produce fresh acid for kerosene and water treating. The can-manufacturing operations will produce and fill about 15,000 5-gal. cans daily.

#### AUSTRIA

##### Export controls

The export of certain Austrian commodities has been made subject to licensing by the Ministry of Commerce. Commodities subject to export licences under the new regulations include, among others, industrial fats and oils, solid mineral fuel, crude oil, non-ferrous metals, iron and steel, chemicals and fertilisers.

## GERMANY

### Plastics industry's expansion

A considerable increase in productive capacity was the keynote of the West German plastics industry during 1955. Further expansion is planned for the current year.

During the first 10 months of 1955, the industry's output rose to about 347,000 tons from 269,000 tons in the same period of 1954, according to figures released by the Chemical Industry Association. For the year as a whole, production is estimated at between 420,000 and 425,000 tons, against 325,000 tons in 1954.

The biggest expansion in productive capacity was noted in polymerisation products, whose output increased by 32½% to 163,000 tons in January/October 1955. Polyvinyl chloride (PVC), polyethylene and polymethacrylene benefited most, in view of the expanding market for these products.

### Scholarships for Burmese

The East German Government have offered a number of scholarships to train Burmese technicians in mining and research institutions and a group of Burmese mining specialists will visit East Germany next month to study mining conditions.

## NETHERLANDS

### Experimental furnace

A new experimental furnace has been put into use by the Royal Netherlands Blast Furnace & Steel Mills at IJmuiden, according to a report from the Board of Trade in London. This furnace is designed to burn coal powder and the experiments are being made by the Foundation of International Flame Research with the assistance of a grant of \$105,000 from the High Authority of the European Coal and Steel Community.

## SPAIN

### Cement project

A firm styled 'Cementos del Cinca S.A.' has been formed with the participation of Hidro Nitro Espanola for the production of cement. Authority has been granted to Ramon Pont to erect a factory at Calaf (Barcelona) for the annual production of 30,000 tons of cement.

## THAILAND

### Cement project

The Irrigation Department placed an order worth U.S. \$2 million with Messrs. Fried Krupp Rhein-Lansen, of Germany, for a cement works to be set up at Tha Klee, near Chainat, to supply the department's cement requirements. Tenders were first invited

### The Leonard Hill Technical Group—February

Articles appearing in some of our associate journals this month include:

**Paint Manufacture**—Stearates for the Paint Industry, 2; Formulation of Industrial Paint Removers; Texture Finishes; Maleic Modified Drying Oils.

**Food Manufacture**—Annual Reviews: Confectionery, Milling, Dairy Products, Vitamins; Vinegar Manufacture; Microscopical Technique for the Food Analyst, 1; The Vacuum Contact Plate Method of Fruit Dehydration, 2.

**Manufacturing Chemist**—Solubilisation with Amphiphilic Compounds, 1; Effect upon Emulsions of the Hydroxy Compounds in Beeswax; Recent Developments in Pharmacognosy; Chromatography—Its Future in Industry; Progress Reports: Therapeutics, Pest Control Chemicals, Perfumery and Essential Oils.

**Fibres**—Variance Length Curves for Jute Yarns: The Use of the Uster Evenness Tester; The Future of Abaca; The Transport Position.

**Petroleum**—European Association of Exploration Geophysicists: Report on Ninth Meeting; Recent Developments in Oil Geology, 1; Corrosion Inhibitors in Petroleum Refining.

**Corrosion Technology**—The Sodium Hydride Process for Descaling Metals; Low-Temperature Corrosion by Flue Gas Condensates.

**Atomics**—Chemical Processing in the Nuclear Power Industry, 1; Reactor Transfer Functions; Barrier Creams in Radiochemical Operations.

**Dairy Engineering**—Cleaning and Sterilising Aluminium Dairy Equipment; Crate Stacking Equipment; Electric Dairy Water Heating and Steam Sterilising Equipment, 1; Developments in Filling Machines.

**World Crops**—Control of the Nun Moth in the Forest of Ebersberg; Control of Game and Vermin Animals; The Work of the Sarawak Department of Agriculture; The Swaziland Irrigation Scheme.

in the summer of 1952, then in 1954 selected firms, which included no British firm, were asked for new quotations. The works are to produce a minimum of 60,000 tons p.a. of Portland cement. The operations which the department has in hand require 30,000 tons p.a. and, if projects which the department has in view materialise, total requirements will rise to 100,000 tons p.a.

## DENMARK

### Chemicals from gas works

The new A. P. Moller pyrolytic gas works in Amager came into operation recently. The works will enable Denmark to be self-sufficient in respect of a number of chemical products which so far have had to be imported.

The Copenhagen Electricity Authority are said to be planning the construction of a power station near the new gas works.

## MALAYA

### Steel mill planned

The first steel mill in Malaya is planned to begin operations in April 1956. The firm is the Singapore Steel & Iron Mills Ltd., and the project is to be completed in three stages in three years. One rolling mill will be installed in the first year and another in the second year; in the third year it is planned to bring an electric furnace into operation for refining steel. The main products will be mild steel, round bars, flat bars, square bars and angle iron and it is estimated that in the first year production should total 3,000 to 4,000 tons, rising to 7,000 tons in the second year and to more than 10,000 in the third year.

## MEXICO

### Change in mining laws sought

Mexico's iron-ore reserves, mainly haematite, have been estimated at 500 million tons, whilst known reserves of coking coal were given as 2,000 million tons. Changes in Mexican mining legislation to make it possible to work these reserves and those of manganese, etc., on an economic basis have been asked for.

### Sulphuric acid

Six new sulphuric acid plants will come into operation in Mexico this year with a total daily production of 170 tons, using Mexican sulphur. Total production in 1956 should therefore reach 175,000 tons, as against 125,000 in 1954. Total consumption in 1954 was 131,550 tons.

## SWEDEN

### Iron and uranium projects

A new mining company, Norrlands Gruv A.B., has been founded at Sundsvall, northern Sweden, to exploit iron ore deposits in the northern provinces of Västerbotten, Norrbotten, Kopparberg and Jämtland. The initial share capital will be Kr. 1.5 million.

Sweden's first atomic fuel factory, which starts operations at the end of the year, will produce annually about 5 tons of highly refined uranium from shale quarried in the Kilberg escarpment near Kvarntorp, central Sweden. The latter has a uranium content of only about 200 g. ton of shale, but extraction is easy and in all may be expected to yield about 1 million tons of uranium.

## NORWAY

### Tube rolling

Norwegian and Netherlands interests have worked out plans for the construction of a tube rolling mill at Mo i Rana, North Norway, at a cost of £5½ million. Production would be about 70,000 tons p.a. and would nearly all be exported. The steel for the tubes would come from the new steel plant at Mo i Rana.

### Aluminium production up

Production of aluminium in Norway in 1955 was 70,000 tons. Production capacity is about 95,000 tons, but was not fully used owing to shortage of hydroelectric power following the very dry summer. There are several projects in hand for increasing production. A S Ardal og Sunndal Verk is to increase production by 36,000 tons, and Elektrokemisk A S is to build a new plant with an output of 22,000 tons p.a. Two other concerns are to increase output by altogether 10,000 tons. In the course of the next five years, total production of aluminium in Norway should therefore be stepped up to about 160,000 tons.

## SOUTH AFRICA

### Imports of drugs, chemicals and fertilisers

During the first five months of 1955 imports were £2 million higher than in the corresponding period of 1954. Most of the increase was in unspecified products, but there were small increases in imports of sulphur, glycerine and a drop of some 8,500 tons in imports of ammonium nitrate. The United Kingdom and the U.S. are equally prominent in this field, but Germany has been gaining ground rapidly.

### Oil blending

A South African correspondent reports that the new blending plant of the Vacuum Oil Co. of South Africa (Pty.) Ltd. at Durban is now in active production. The capacity of the plant on a one-shift basis is equal to about 8 million gal. p.a. of high-grade lubricating oils.

The new plant, which was completed about the middle of 1955 at a cost of some £200,000, includes a special blender with a five-panel controller powered by a master drive so that it is possible for three different base stocks and two additives to be mixed at one time to rigid specifications. If any constituent of any desired blend does not flow in the proper proportion, the automatic controls shut down the unit at once. Contamination

in the blender is avoided by the provision of a blow-back system. The blended products are run into the containers through two separate filling lines.

## CHILE

### Metallurgical coke

A plant for the production of metallurgical coke is to be erected by the Corporacion de Fomento de la Produccion near Valdivia utilising the *Convertol* process. The necessary installation is being constructed by the German firms Krupp and Otto, of Essen.

### Soda

A new company, the Sociedad Quimica Industrial Caupolican Ltda., is to be formed under the aegis of the Corporacion de Fomento de la Produccion for making different forms of soda, using nitrate of soda. The Jourdan system will be employed whereby caustic soda, carbonate of soda and nitric acid are obtained.

## INDIA

### Polystyrene factory

A factory for the manufacture of polystyrene with a rated annual capacity of 6 million lb. is to be set up near Bombay by Polychem Ltd. The plant is expected to go into production in October 1956.

A spokesman of the company said that the production would be more than sufficient to meet the requirements for polystyrene of the entire Indian plastics moulding industry.

The company, which has been sponsored by the Kilachand Devechand Co., of Bombay, and the Dow Chemical Co., U.S., will have an authorised capital of 15 million rupees, with an issued and paid-up capital of 6 million rupees. Dow is expected to invest 1 million rupees and has agreed to supervise the erection of the factory and to bring its production up to normal capacity. It will also train Indian technicians in the United States.

## TANGANYIKA

### Limestone discovery

Valuable limestone deposits have been discovered in a hill 13 miles north-west of Dar-es-Salaam. Business men in America, Britain and several other countries are showing keen interest in the discovery for the production of cement. Pure limestone has been proved to extend to the maximum depth of 45 ft., which indicates a reserve in the region of 21 million tons or possibly more.

## UNITED STATES

### Chemical company's projects

The American Cyanamid Co. is planning to build new plants for chemical production at a cost of between \$40 million and \$50 million.

Plans are being completed for a project which aims at doubling the acrylonitrile capacity of the company's Fortier, La., plant which at present produces 50 million lb. p.a. of this product. This reflects increasing demand from the synthetic fibre industry.

A plant for the manufacture of acrylamide, a new U.S. chemical used by the mining and paper industries, is also under consideration.

Market tests are being made of the company's synthetic fibre called *Creslan*, but no decision has yet been made as to when commercial production is to start.

A new plant at Brewster, Fla., is being built to make 200,000 tons p.a. of triple superphosphate. Other plant will also be built to make some new thermoplastic moulding compounds using acrylonitrile and acetylene as raw materials.

### Chemical milling of magnesium

The Dow Chemical Co. and Turco Products are to undertake the development of a process for the chemical milling of magnesium. This is said to be faster and more flexible than conventional milling methods, while the equipment required is cheaper.

A joint announcement by the companies said an exchange of technical and processing information had already resulted in the production of a highly efficient etching solution for magnesium milling. Dow said the chemical milling method had achieved tolerances as close as  $\pm 0.003$  in.

### Open-pit mining of sodium borate

Open-pit mining, an ultimate step in the \$18-million development of the world's only known sodium borate deposit, has started at Boron, California, after 30 years of shaft tunnel operation. The rest of the project, including refining and concentrating plants, is scheduled for completion in 1957.

The mine is that of the Pacific Coast Borax Co., a division of Borax Consolidated Ltd.

## RUMANIA

### Hungarians run pilot plant

A 'colony' of Hungarian technicians has settled in Bucharest to work on a chemical test plant, now nearly completed, at what will be the sending end of a 250-mile pipeline to



carry methane gas to Hungary's biggest chemical combine now being erected at Tiszapalkonya. Every day they travel 10 miles to the village of Buciumen, where the factory is being built on the banks of the Colentina.

Part of the plant is already producing an acetylene-synthesis gas mixture. The acetone section is to start experimental production in the spring.

The factory is being built to plans of Hungarian scientists, and most of the equipment is made in Hungary. By the end of this year it is expected the whole factory will be producing chemical products for Rumanian industry, and the experiences gained will be applied to the much bigger Hungarian combine, which is to produce raw materials for the plastics industry.

Two methods are being tested at the

Rumanian plant. One is the partial-oxidation of the methane gas to produce a mixture containing acetylene and synthesis gas, from which acetone can be manufactured. The other method is hypersorption, worked out by the Hungarian Oil and Natural Gas Experimental Institute, to break down the mixture into acetylene and synthesis gas.

The Tiszapalkonya combine on the banks of the Tisza in north-east Hungary is the biggest investment of the current Hungarian five-year plan. This year alone, it is announced, more than £2 million is being spent on the foundations of the plant itself, the 200,000-kw. power station which will serve it, and the new town which will house the thousands of workers to be employed there.

## Recent Publications

**Instruments.** From C. F. Casella & Co. Ltd. comes a 176-page catalogue (No. 808) describing and illustrating a range of thermometers, hydrometers and scientific instruments.

**Space-heating plant,** featured in an illustrated pamphlet from Weather-foil Heating Systems Ltd., is stated to be particularly well suited to heating warehouses, factories, rolling mills, etc.

**Fine screening.** The Symons 'V' screen, which multiplies screening action by combining centrifugal and gravitational forces, is described and illustrated in bulletin 243 from Nordberg Manufacturing Co., U.S. The screen is specifically designed for fine screening, for sizing, dewatering, dedusting, cleaning and washing.

**Remote control.** Two new leaflets have come from Londex Ltd. The first deals with the RFT synchronous process timer, which is for panel mounting, is adjustable, and resets automatically; the second deals with a single-hole-fixing signal lamp fitting.

**Aliphatic amines.** The Chemical Division of Armour & Co. Ltd. have published an 8-page booklet on *Armeens*, which is their registered trade mark for long-chain aliphatic amines. The booklet covers both primary amines  $RNH_2$  and secondary amines  $R_2NH$ . There are two tables which give the chemical composition and principal physical and chemical characteristics of these amines.

**Vacuum equipment.** A number of new leaflets have been issued by Edwards High Vacuum Ltd. to supplement their catalogue on vacuum equipment. One of these deals with a new vacuum gauge, the *Philani*, while another covers an ionisation gauge control unit and the complete range of gauge heads. A third publication is an up-to-date version of the gauge summary D 111/2. On the subject of vacuum accessories, there are leaflets dealing with 'O'-ring vacuum seals; vacuum unions and flanges; fine control needle valves; and magnetic vacuum valves. Vacuum coating units 6E and 12E are described, along with their accessories, in two further leaflets. The deposition of metallic films under vacuum requires very careful selection of the right materials and choice of suitable lacquers, and one of the latest publications gives details of the evaporation filaments, baskets and boats which Edwards supply. Yet another provides information on suitable evaporation materials and recommended suppliers.

## MEETINGS

### Institution of Chemical Engineers

February 21. 'Heat Transfer Media for the Control of Reactions at Elevated Temperatures,' by P. A. Rottenburg, 7 p.m., Grosvenor Hotel, Chester.

February 25. Symposium on 'The Modern Methods of Counter-Current Vapour-Liquid Contacting,' 2 p.m., College of Technology, Manchester.

February 28. 'A Case Study of the Expansion of a Fine-Chemical Plant,' by B. L. Budd and J. S. Brough, 7 p.m., College of Technology, Manchester.

March 6. 'The Development of Mixer-Settler Equipment for Liquid-Liquid Extraction,' by B. T. Bell and F. Roberts, 5.30 p.m., Geological Society, Burlington House, London, W.1.

March 7. 'Recent Developments and Future Trends in Petroleum Refining Technology,' by W. Harvey, 6.30 p.m., Midlands Institute, Birmingham.

### Institute of Fuel

February 15. 'The Oil-from-Coal Project of the South African Government,' by P. E. Rousseau, 5.30 p.m., Institution of Civil Engineers, Great George Street, London, S.W.1.

February 15. 'Solid-fuel-burning Gas Turbines,' by J. D. Thorn, 2.30 p.m., Royal Victoria Hotel, Sheffield.

February 23. 'Coal Preparation and Evaluation,' by J. K. Matthews, 7 p.m., Liverpool Engineering Society, 9 The Temple, Dale Street, Liverpool.

February 23. 'Hot-blast Cupola Furnaces,' by W. J. Driscoll, 6 p.m.,

James Watt Institute, Great Charles Street, Birmingham 3.

### Society of Chemical Industry (Chemical Engineering Group)

February 14. 'Manufacture of Phosphorus,' by F. B. Shepherd, 5.30 p.m., Geological Society, Burlington House, London, W.1.

### Incorporated Plant Engineers

February 14. 'Application of Maintenance of Centrifugal Pumps,' by G. O. Stevenson and A. J. Clements, 7.15 p.m., Engineers' Club, Albert Square, Manchester.

February 28. 'Oil Firing,' by Mr. Milne, 7.15 p.m., South Wales Engineers' Institute, Park Place, Cardiff.

### Chemical Society

February 21. 'The Fischer-Tropsch Synthesis: A Challenge to the Physical Chemist,' by Prof. C. Kemball, 7.15 p.m., Queen's University, Belfast. Joint meeting with the R.I.C. and S.C.I.

February 23. 'Some Developments in the Study of Physical Adsorption,' by Prof. D. H. Everett, M.B.E., 7.30 p.m., North British Station Hotel, Edinburgh. Joint meeting with the R.I.C. and S.C.I.

March 5. 'New Developments in Inorganic Chemistry,' by Prof. R. S. Nyholm, 6.30 p.m., The University, Leeds.

March 8. 'Chemical Engineering and its Place in the University,' by Prof. K. G. Denbigh, 5 p.m., Queen's College, Dundee.

# WHAT'S NEWS *about*

This illustrated report on recent developments is associated with a reader service that is operated free of charge by our Enquiry Bureau. Each item appearing in these pages has a reference number appended to it; to obtain more information, fill in one of the attached postcards, giving the appropriate reference number(s), and post the card (no stamp required in the United Kingdom).

## Portable cooler handles 500 gal./hr.

A new range of continuous and totally enclosed *Crystal* water coolers, delivering water under pressure, is being manufactured in the United Kingdom. Suitable for any manufacturing process where a continuous water-cooling system is essential, the coolers are mounted on one base and are completely portable. This feature cuts down installation time and caters for quick and simple relocation.

The range is based on a cylinder unit having a capacity of 50,000 B.Th.U. Three models are available, ranging up to the three-cylinder model having a capacity of 150,000 B.Th.U. The three-cylinder model will cool 500 gal./hr. of water from 70 to 40°F. continuously, or 250 gal./hr. from 100 to 40°F.

A thermostatic control, common to all models, has been devised to maintain constant temperature control of any water drawn out of the system. Another important control is a back-pressure regulator which limits the suction pressure in the evaporator to between 28 and 30°F. in order to avoid any possibility of freezing.

The 15-h.p. compressor of the three-cylinder model is controlled by the thermostat and low-pressure cut-out so that, in the event of either the thermostat or suction control valve closing, the compressor will stop.

All models are mounted on a base to form a self-contained unit, and are ideal for shipping or transporting. The coolers are made in aluminium throughout, or stainless steel with chrome-steel headers. The aluminium models have been developed because they show slight reduction in price and materials are more easily obtainable.

A range of syrup coolers is also manufactured and of particular interest

are the chilled-water syrup coolers available in a standard capacity range of 25, 30, 40, 50, 60, 75 and 100 gal./hr.

One model is self-contained, with refrigeration. This has a capacity of 45 gal./hr. with a temperature range

of from 85 to 40°F. (20,000 B.Th.U.) and is operated by a 3-h.p. air-cooled refrigerating compressor.

All parts of the coolers in contact with syrup are of stainless steel, and headers are quickly removable for cleaning.

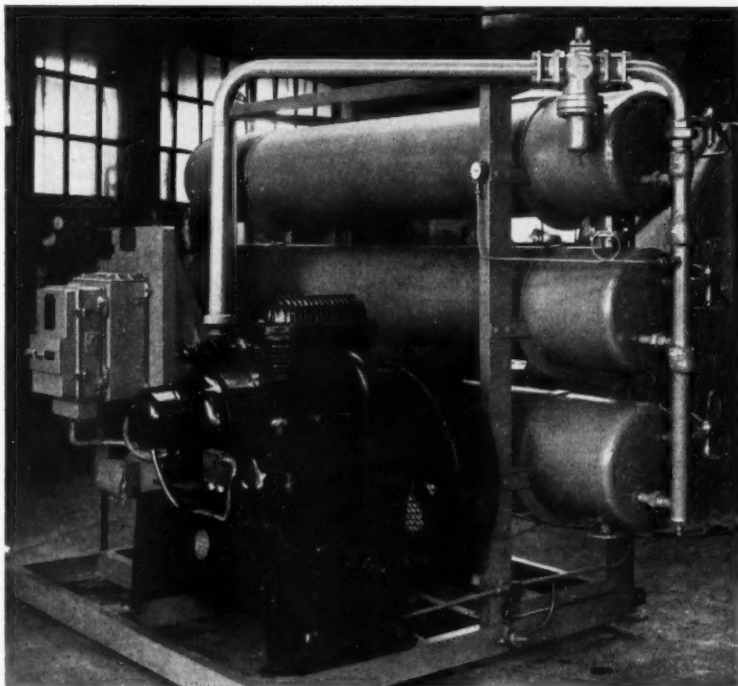
CPE 171

## Unit construction of process plants

A simple and economical method of constructing chemical process plants, using standardised components, has been evolved by a British firm, who are manufacturing equipment for this purpose.

The idea is based on the fact that all chemical process plants consist of

a series of special vessels and ancillaries connected together in such a way to satisfy process requirements. A large range of components has been made available from which units of the type and capacity needed can be selected. It is stated that the application of this method is almost unlimited



Three-cylinder water cooler.

## C.P.E.'S MONTHLY REPORT AND READER SERVICE

CHEMICAL & PROCESS ENGINEERING, February 1956

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where chemical or physical operations are part of an overall process requirement.

The components, whatever their type or size, are constructed so that they can be assembled on a chassis, and are completely interchangeable with one another. The chassis on which the various components are assembled consists of three prefabricated horizontal frames, connected together with braced stanchions, from which one-, two- or three-tier structures can be obtained.

For the simple requirements, it is possible that only one chassis assembly will be required, but with complicated processes, involving many different stages, several assemblies can be married together to form an efficient and neat plant.

With technical requirements decided, it is necessary only to select equipment of suitable type and capacity from the standard range. Thus wasting of valuable drawing-office time and manufacturing problems are avoided and the plant can be built and commissioned quickly.

Not only are the various vessels and ancillaries catered for, but also such items as switchgear, with prefabricated cable, and instruments, which are all in a weather-proof case, ready for mounting on the chassis. When all the components are assembled, it is only necessary to couple the main service supply lines and the plant is ready for operation.

With the assembling of components on a standard chassis, the overall plant layout is predetermined. This enables

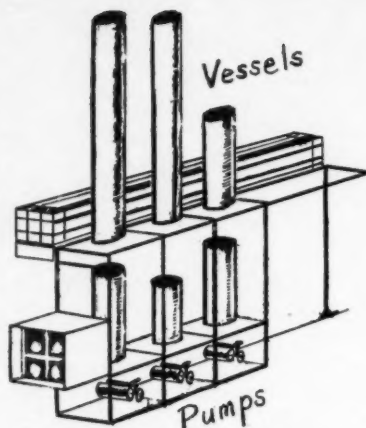


Diagram illustrating the idea of unit construction of process plants.

a location to be decided upon before even the drawings are started. Other advantages from the design point of view include compactness, accessibility of the components, simplified maintenance and the absence of any need for special foundations.

From the point of view of the process, an outstanding advantage is that process variations are easily made, as it is only necessary to modify the pipe flows and possibly some of the internals of the components. Capacity variations are also very simple, while, if it should become desirable to discontinue the process for which a plant is installed, there is no question of complete capital loss. For pilot-plant work, a unit with small components can be quickly assembled and is easily changed about afterwards.

CPE 172

## Concrete repair material lines process vessels too

The *Gunit* process, which has been used in industry for a variety of purposes such as, for example, the repair of reinforced concrete factories which have deteriorated through weathering and rusting of the reinforcement, has also been used for the lining and repair of process tanks, the encasement of steel work as a protection against corrosion or attack by dilute acid fumes, and the lining of steel bunkers and chimneys.

It has been found that the reinforced *Gunit* lining of a steel process vessel can be carried out for a fraction of the cost of the previous linings. On a large chemical works where this was done it had previously been the custom to provide a form of lining which proved to be extremely costly

to renew, as had been frequently necessary owing to corrosion.

*Gunit* consists of a good quality selected sand, graded from about  $\frac{3}{16}$  in. down to fines, mixed with cement. The peculiar properties of high density and hardness are achieved by the method of application of the *Gunit*. The materials are carried in a dry form from a cement gun up to the nozzle through a material hose at a pressure of 30 to 35 p.s.i. At the nozzle, water is admitted through an atomiser, and the skilled operator can control the amount admitted so that the mixture leaves the nozzle jet with only the minimum amount of water required for hydration of the cement. Thus there is no excess water to dry out and leave pores in the *Gunit*.

Furthermore, the material is projected with such force that, as it is being built up on the surface, it is being continually tamped or rammed by the succeeding particles. The material can be applied in layers of 1-in. thickness to overhead or vertical surfaces quite safely without any danger of slumping. Any desired thickness may be built up, but it is usual to allow an interval of not less than 12 hr. between the application of each 1-in. layer to ensure that the material will not sag under its own weight before setting.

The cement used in the *Gunit* mix is generally ordinary Portland cement, but, in cases where there may be chemical attack, aluminous cement is sometimes to be preferred. Thus, for the lining of steel chimneys, a mixture of aluminous cement and high-quality sand is used and the *Gunit* thickness is generally  $1\frac{1}{2}$  in. or 2 in. The reason is, of course, that, when the hot gases attain a certain height in the chimney, they cool down to the dewpoint and moisture containing dilute sulphuric acid condenses on the inner surface of the chimney. The aluminous cement *Gunit* lining is resistant to this attack, and chimneys so lined have been in constant use for many years.

CPE 173

## Synthetic rubber compound protects equipment

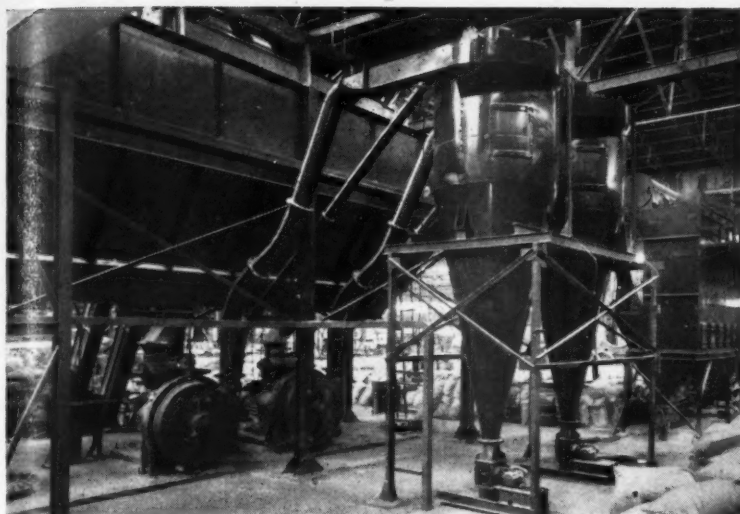
Larger quantities of the new synthetic rubber compound *Hypalon* will shortly become available in the United Kingdom as the result of plans for a new factory announced by its American producers.

The compound can be brushed or sprayed on to any material which is likely to suffer from atmospheric or chemical corrosion. It forms a protective coating which, the makers state, remains intact and flexible at high and low temperatures and provides complete protection against ozone, sunlight, bad weather and many chemicals. It will adhere to natural rubber, synthetic fibres, wood, stone and metal and shows unusually good resistance to scuffing and abrasion.

The coating is primarily intended for industrial equipment such as conveyor belts, cables and hoses, but the wide range of colours available enables it to be used also on floor tiles, footwear, upholstery fabrics and many other household goods.

CPE 174





An 'Atritor' installation for grinding sodium tripolyphosphate in the large chemical works of Albright & Wilson Ltd.

## Controlled drying and grinding with novel pulveriser

A self-contained unit which pulverises, dries and delivers the product in one operation is known as the *Atritor* because it pulverises entirely by attrition. It consists principally of a feeder, pulveriser and fan, and functions as follows.

The material is fed into the hopper on the machine and descends on to a horizontal rotating disc. The spreading of the material over the disc is controlled by a sleeve which is adjustable for height, and the feed to the machine is regulated by a knife which scrapes the material off the disc. The position of the knife is controlled by a handwheel.

The feeder is specially designed to obtain fine regulation. No jamming is possible and large detachable covers are fitted for inspection or cleaning purposes when necessary.

Passing down the chute, raw material then enters the pulverising compartment on the first-effect side where it is subjected to the disintegrating effect caused by a number of hammer segments.

After this treatment the material is much reduced in size and can be carried over the periphery of the rotor into the attrition zone by the conveying air. In this zone the movement of the particles is most complex. They are carried inwards to the centre and outwards again to the periphery and back again until the particles are reduced to the correct size. This intense

turbulence is created by a series of large impellers mounted on the rotor, which sets up terrific eddies and vortices which rub the particles together. The fixed pegs or interrupters facilitate and accelerate the creation of these vortices and also prevent the gyration of the dust cloud with the rotor, thus permitting the impellers to maintain their full effectiveness.

When the particles have become superfine they are gradually drawn out of this turbulence towards the centre and the 'eye' of the fan where they are intercepted by a device termed a rejector. The function of the rejector

is to prevent any oversize particles being drawn by the fan from the pulverising zone. The rejector consists of one or more spoon-shaped arms rotating with the shaft and rotor. These arms are set closely adjacent to the eye of the diaphragm which separates the pulverising and fan compartments. The number of these arms governs the size or mass of the particle ultimately required. It will be readily appreciated that if the particle is large it will travel much more slowly on the air stream than a small particle, since all particles are sustained by the slip of the air passing them. The coarser particles are intercepted by the rejector and returned into the attrition zone, while the finer particles pass to the fan.

The drying of the material is effected by a supply of hot air, from a coke or oil-fired stove, which passes through the *Atritor* with the material. As the *Atritor* contains only a few pounds of material at any moment and the whole of this material is in circulation, the surface exposed to the drying influence of the hot air or gas is enormous.

The machine requires a minimum of attention and can be operated entirely by unskilled labour.

CPE 175

## Spray drying

A Danish company is offering a spray-drying plant which is stated to be suitable for the drying of milk, coffee extract, cheese, eggs, bananas, yeast, blood, soap, synthetic washing agents, tannin extract, etc., and also for heat-sensitive materials.

In this plant, the drying chamber is constructed as a cylinder with a slightly conical bottom from which the dried product is continuously removed by means of a rotating, pneumatic discharger. Any accumulation of the dried product in the chamber is thereby avoided. All the powder produced leaves the chamber together with the drying air and is separated from the air in cyclones.

The atomiser is of simple construction and can be stripped down and reassembled even by unskilled workers, since it contains no gear, oil pumps or fragile parts—in operation this has the additional advantage of ensuring against sudden stoppages. All surfaces contacting the liquid are of stainless steel and are easily accessible for cleaning. The atomiser runs smoothly and it is not necessary to bolt it to the foundation; instead it simply rests on a rubber ring.

The *Anhydro* spray-drying system,

### FIRE-RESISTANT SYNTHETIC OILS

The American manufacturers of *Cellulube* fire-resistant synthetic oils announce that the series has been expanded to include hydraulic fluids and lubricants in six controlled viscosities. These fluids, which the makers describe as straight phosphate esters, are now available in viscosity ranges of 90, 150, 220, 300, 500 and 1,000 SUS at 100°F.

Addition of these viscosity ranges has broadened the use potential for *Cellulubes* in industries where fire resistance in functional fluids is a significant safety factor.

CPE 176

as it is called, is available in three types, the most commonly used of which is distinguished by the steam heating of the air. Another type has an indirectly oil-fired heater; this also is a general-purpose type and has the special advantage of being able to operate with higher temperatures, whereby particularly economical drying is obtained.

The third type is a high-temperature plant which is especially suitable for the drying of technical products and is only exceptionally used for food-stuffs. The drying medium consists of a mixture of the combustion gases from an oil burner and air.

A recent introduction is a laboratory spray drier which the makers describe as an accurate copy of their industrial plants. The laboratory model is operated with electricity only, being provided with a system of electrical heating wires. The speed of the atomiser can be varied up to 50,000 r.p.m. It is all made of stainless steel and is very easy to strip down and clean.

CPE 177

## Fume scrubbing and absorption tower

A fume scrubbing and absorption tower which is claimed to be 100% efficient in the scrubbing of air saturated with sulphuric and hydrochloric acid, and 99.9% efficient with nitric acid, has recently been produced.

The *Turbro* fume scrubbing and absorption tower has been specially designed for the elimination of sulphuric, hydrochloric or nitric acid fumes discharged in considerable quantities from various chemical and industrial processes. The S/1 unit is capable of scrubbing fumes from 14 to 20 l./hr. of evaporated acid; larger towers are in course of construction for many varied fume scrubbing requirements.

The tower and internal components are constructed entirely from B.X. *Cobex* rigid vinyl sheet, and it is claimed that they are therefore unaffected by the strongest of acids. The tower can be supplied with a special fume cabinet or, alternatively, as a single unit for incorporation into existing equipment. The S/1 tower is approximately 8-ft. high  $\times$  2-ft. square.

The complete removal of the gases is carried out in three stages. The first is by absorption of greater part of the gases in liquid, after which suspended globules of moisture are

eliminated in a special eliminator section and, finally, any small traces of gases left after these two processes are completely neutralised by a chemical reagent contained in a tray.

CPE 178

## Regulus acid pumps

Acid pumps are being marketed which are stated to be of wide application where sulphuric acid lifting is concerned. They are of a heavy design, and are constructed in regulus metal throughout—with the exception of the shaft, which is of stainless steel or *Monel* metal according to the strength of the acid required to be pumped. They are made in self-contained units, combined with electric motor, and in use for sulphuric acid up to 40-ft. lift.

CPE 179



Fume scrubbing and absorption tower.

## BRITISH FLEXIBLE PIPING FOR U.S.

A British firm of thermoplastics designers and manufacturers were recently able to supply an American company with a material that they were unable to obtain in the U.S. The American firm, who are concerned with rocket engine development and research, could not obtain a type of flexible piping suitable for special purposes in rocket propulsion, so, as the project in hand was vitally urgent, British assistance was sought. A British firm was able to produce the material required in a matter of hours and ship it by air to the States.

The material needed was an extremely flexible type of piping which would be resistant to most types of corrosive acids and which could be twisted and bent without reducing the internal bore. The answer was a special type of PVC-covered steel-spring industrial tubing in uninterrupted lengths known as *Armourvin*. So, after an exchange of priority cables between Britain and the U.S., quantities of  $\frac{3}{4}$ -in.-bore *Armourvin* were on their way by air to assist with America's rocket development programme.

CPE 180

## One-component epoxy resins

As a means of simplifying the use of epoxy casting resins in the electronic industry, a series of single-component formulations requiring no catalysts or hardeners are now offered by an American company. The *Aritemp* resins are free-flowing powders which, on melting, form low-viscosity solutions permitting the impregnation and sealing of complex electronic components. It is claimed that plant procedure is simplified and the possibility of errors in formulation is avoided by the use of single-component systems. An error which could result in the loss or necessity of salvaging valuable equipment is prevented.

A unique property of these resins is that they become rubbery rather than brittle at high temperatures, thus enabling their use with small wires and fragile components which are prone to damage. Potted components may be temperature-cycled from -60 to 150°C. without the development of cracks or dielectric losses.

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